

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 68 Number 4

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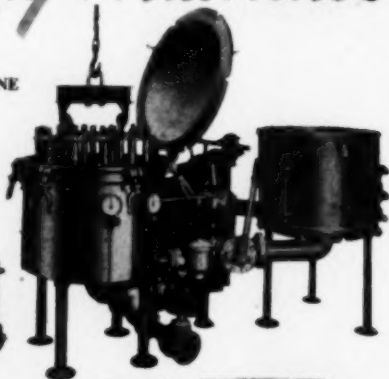
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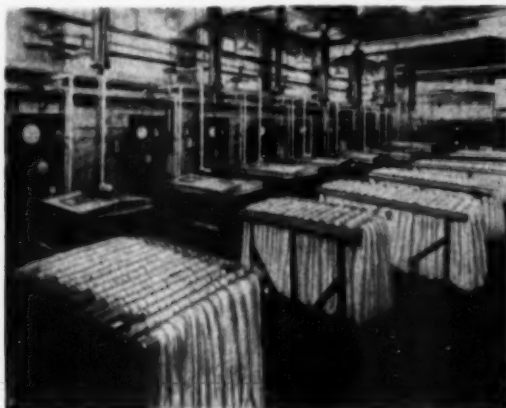
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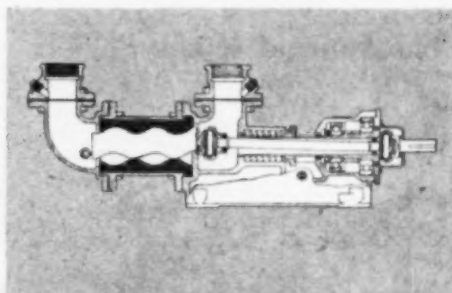
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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

A New Method for the Dyeing of Mineral Khaki

E. H. Daruwalla and G. M. Nabar

Quantitative Estimation of Eulan CN, Mitin FF, and Lanoc CN in Wool

R. S. Hartley, F. F. Elsworth, P. G. Midgley, and J. Barritt

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THE JOURNAL

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Volume 68—Number 4

APRIL 1952

Issued Monthly

COMMUNICATIONS

Package Dyeing

I—A Theoretical Model and its Relation to Technical Practice

J. BOULTON and J. CRANK

The general problem of package dyeing is outlined and a theoretical model is discussed in detail. This is the first step in an attempt to gain a fuller understanding of the basic processes operating within the package. The model is shown to lead to results which are in general agreement with observations from standard dyeing practice. The necessity of avoiding high exhaustion is confirmed by the model, and it is emphasised that level dyeing is most likely to be secured by rapid flow and low exhaustion. A number of ways in which the practical dyer may seek to produce uniformly dyed packages are examined critically from the point of view of their feasibility in commercial dyeing.

THE GENERAL PROBLEM OF PACKAGE DYEING

An increasing quantity of fibres and yarn of all kinds is now dyed in package dyeing machines, in which a circulating dye liquor is pumped through a stationary package and is progressively exhausted as the substantive dye is absorbed by the yarn. The principles of package dyeing apply to all prepared packages such as cops and cheeses of cotton, wool, and rayon, and to box-spun cakes of continuous-filament viscose rayon, all of which are dyed in machines such as the Longclose or Courtaulds types, where separate packages are mounted individually. They apply also to packages, such as loose fibre, packed hanks, and sliver dyed as an annular wall in the Obermaier type, or as a bed in the flat-container circulating machine as used for loose wool, and also to tops in the can-type container. The dyer's task is to ensure that, when the overall appearance of his material matches his pattern, dye is uniformly distributed throughout the whole mass of the package. Level dyeing is easily attained with Class A direct dyes on cellulosic packages or with the levelling type of acid dyes on wool packages, but it is not attained in a commercially reasonable time with strong-alkali leuco vat dyes on cellulosic packages or with some aggregated-type acid dyes on wool tops.

Some of the problems encountered in package dyeing have been described elsewhere¹⁻⁵. The basic difficulty is that many dyes, especially under conditions of high dyebath exhaustion, are absorbed rapidly on to the substrate in regions most accessible to the flowing liquor, causing local over-dyeing, removal of which is either tedious or impossible.

Perfect levelness may be impossible, because the fibre to be dyed has intrinsic dye-absorption

properties which are non-uniform or, in the case of yarns spun from fibres, because of non-uniform spinning. But this is not the major problem; with natural fibres and with any fibre in the loose or slubbing state, such imperfections are levelled out by blending in the dyed state which precedes textile manufacture. In the case of continuous-spun man-made filaments, especially the older established regenerated celluloses and cellulose esters, dye-absorption properties throughout the length of an extrusion-spun unit package have now attained a relatively high degree of uniformity. The major difficulties are those associated with the properties of the dyebath and with the conditions of dyeing, including the mechanical and geometrical properties of the particular fibre or yarn package and the kind of machine used. By properties of the dyebath are meant the combined influences exerted by the precise characteristics of the dye or dyes, and the influence upon these of such variables as electrolyte concentration, temperature, and liquor length (volume ratio). By package properties are meant the thickness and density of the wall or bed, cone or cheese, or cake, of fibre or yarn which is to be penetrated and the uniformity of its disposition in the machine.

In this paper we shall assume that the intrinsic dye-absorption properties are, in fact, uniform and that unevenness results from the remaining causes entirely, and we shall in the first place be concerned mainly with the properties of the dye itself and the fundamentals of its distribution throughout a model package.

Although the practical dyer is well aware of the difficulties, it is not easy for him to study the basic causes of the trouble, and in fact he may not always be certain whether the difficulties are due to the

yarn or to his dyeing conditions. An investigation under laboratory conditions is more likely to reveal fundamental causes and is an essential feature of any attempt to improve dyeing technique, but even in the laboratory, with so many interdependent variables involved, it is not easy to plan experiments in which only one variable is changed at once or to sort out the results in the end. It seems worth while, therefore, to try to set up a theoretical model of package dyeing in order to get some insight into the basic processes operating within the package as the liquor circulates and to isolate the essential variables of the problem. Such a model is described in this paper. It leads to results which are in general agreement with observations from standard dyeing practice, and reveals some of the fundamental causes of troublesome effects observed in practice.

In this first paper of the series the model is described in detail, and the results derived from it are discussed in general terms. In later papers results calculated from the model will be compared quantitatively with experimental data for different dyes in the hope that the points of agreement and discrepancy will yield more information about the processes involved in package dyeing.

THE THEORETICAL MODEL

In order to keep the model as simple as possible we shall consider a package in the form of a cylinder through which the dye liquor flows from one end to the other (Fig. 1). The package is considered to be uniformly constructed throughout, there are no variations of yarn properties through the package, and the flow of liquor is everywhere uniform.

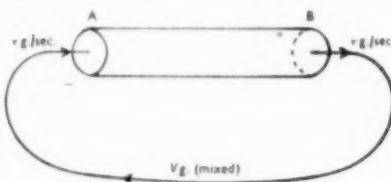


FIG. 1

Suppose that v g. of liquor flows from the dyebath through the end A and returns to the bath through the end B each second, and that the liquor in the bath is always perfectly mixed. The total mass of liquor in the bath is V g.

As the liquor flows through the package two processes occur—

- There is an exchange of dye between the liquor and the yarn at each point within the package.
- As a result of this, the concentration of dye in the liquor changes as the liquor flows through the package; e.g. in the early stages the liquor is progressively exhausted as it flows through.

Considering (a) first, it is clear that this rate of exchange depends on the concentration C of dye in the liquor in any element of package at any time (expressed in grams per gram of liquor) and on

the concentration M of dye on the yarn in the same element (expressed in grams per gram of yarn). We have taken the rate of exchange of dye between liquor and yarn to be related to these two quantities in the simplest possible way which has any useful meaning—

$$\text{Rate of uptake of dye by 1 g. of yarn at any point} = K(E_C - M) \quad (i)$$

(M = amount of dye actually on the yarn; E_C = amount of dye which 1 g. of yarn could take up if left long enough to reach equilibrium with the liquor, of concentration C , in that region of the package). Thus M , C , and E_C vary from point to point in the package and with time. E_C is related to C through the usual absorption isotherm, and we have used the Freundlich expression—

$$E_C = RC^{\frac{1}{n}} \quad (ii)$$

to represent this relation, R being a constant.

Here K is a rate constant for the exchange of dye between the liquor and the yarn and *vice versa* within the package. It is hoped to relate, later, this constant to the diffusion properties of the dye within the individual filaments of yarn or to the rate of uptake of dye on the surface of the filaments. For the moment, however, we shall consider it to be simply an empirical rate constant. We assume throughout this paper that dye has unrestricted access to the surface of a fibre, which means that there is no appreciable resistance to transfer in the liquid phase. To allow for such resistance K would have to be a function of speed of flow. We shall not attempt to include this possibility in our present equations. Subsequent work will show to what extent our assumption is justified. We may note that the expression (i) can represent dyeing of the yarn ($E_C > M$) and washing off ($E_C < M$).

Next we need to express the way in which dye concentration in the liquor changes with the distance through the package. To do this we consider the element of the package between two planes at distances x_1 , x_2 from the end A (Fig. 2). Let the dye concentration in the liquor at x_1 , x_2 be C_1 , C_2 g./g. liquor. Then, since v g. liquor crosses the section at x_1 every second and each gram contains C_1 g. of dye—

$$\text{Amount of dye entering the element} = vC_1 \text{ g./sec.} \quad (iii)$$

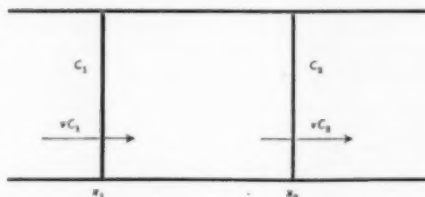


FIG. 2

Similarly—

$$\text{Amount of dye leaving through the plane } x_2 = vC_2 \text{ g./sec.} \quad (iiib)$$

Thus in passing through this element of package the liquor loses $v(C_1 - C_2)$ g. of dye each second, and we can equate this to the amount of dye taken

up by the yarn in the element per second. Thus—

- Dye given up by the liquor per second
 = $v(C_1 - C_2)$
 = Increase in amount of dye on yarn per second
 = Increase in M per second \times Weight of yarn in element
 = Increase in M per second $\times W(x_2 - x_1)$ (iv)

where W is the weight of yarn per unit length of package.

In deriving the relationship (iv) we have assumed that any transfer of dye along the length of the package by diffusion is negligibly slow compared with the rate of mechanical flow, and that the total amount of dye in the liquor in any element of package is small compared with that on the yarn in the same element. The first is a better assumption for slowly levelling dyes (Classes B and C) than for Class A dyes, though it seems unlikely to introduce serious error even for the fastest Class A dyes. The second assumption is more correct the less permeable the package and under conditions of high exhaustion. Some exploratory calculations on packages containing different amounts of occluded water between the individual threads of yarn suggest that errors arising from this second assumption will not seriously modify the calculated results for any case of practical importance.

EQUATIONS DESCRIBING THE MODEL

The above model can be expressed in terms of two partial differential equations—

$$\frac{\partial M}{\partial t} = K(E_C - M) \quad (v)$$

$$-\frac{\partial C}{\partial x} = \frac{WK}{v}(E_C - M) \quad (vi)$$

These differential equations are the counterparts of equations (i) and (iv) above, and are derived in the same way by considering an elementary cell of length δx instead of the finite cell of Fig. 2. Equation (v) expresses the rate of increase of amount of dye on the yarn at any point in the package, and (vi) the concentration gradient in the liquor within the package. It is not possible to write down general solutions of these equations, but numerical solutions for any required set of conditions can be obtained using step-by-step methods of integration. Solutions of this kind have been obtained subject always to two conditions—

(a) There is no dye on the yarn initially before dyeing commences. This is expressed mathematically—

$$M = 0, \quad 0 < x < l, \quad t = 0 \quad (vii)$$

where l is the total length of the package.

(b) The total amount of dye on the yarn and in the bath remains constant during dyeing and is equal to the amount in the bath initially—

$$VC_{\infty} = VC_0 + \int_0^l WM \, dx \quad (viii)$$

because the concentration of dye in the bath (assumed uniform throughout the bath) is equal to the concentration of dye in the liquor when it enters the package at A ($x = 0$). Here C_0 is the dyebath concentration at the commencement of dyeing.

The equations can be simplified and the essential parameters of the problem isolated by introducing the following non-dimensional variables—

$$T = Kt$$

$$X = \frac{WK}{v} \cdot \frac{RC_0^{\frac{1}{2}}}{C_0} x$$

$$c = \frac{C}{C_0}$$

$$m = \frac{M}{RC_0^{\frac{1}{2}}} \quad (ix)$$

In terms of these variables equations (ii), (v), (vi), (vii), and (viii) reduce to—

$$\frac{\partial m}{\partial T} = c^{\frac{1}{2}} - m \quad (x)$$

$$\frac{\partial c}{\partial X} = -(c^{\frac{1}{2}} - m) \quad (xi)$$

$$c = 1 - \frac{v}{KV} \int_0^X m \, dX \quad (xii)$$

$$m = 0, \quad 0 < X < X_l, \quad T = 0 \quad (xiii)$$

where X_l is the length of the package in the new variables and is given by putting $x = l$ in the expression for X (ix), so that—

$$X_l = \frac{WK}{v} \cdot \frac{RC_0^{\frac{1}{2}}}{C_0} l \quad (xiv)$$

This quantity X_l is related to the conditions of dyeing in the following way. In the final equilibrium state the concentration of dye on the yarn is everywhere the same and equal to M_{∞} , and in the liquor the concentration is uniform and denoted by C_{∞} . In this case on dividing each side of (viii) by VC_0 we obtain immediately—

$$c_{\infty} = 1 - \frac{WlM_{\infty}}{VC_0} = 1 - \text{Final fractional exhaustion} \quad (xv)$$

from which c_{∞} is determined for a given exhaustion. Also—

$$M_{\infty} = RC_0^{\frac{1}{2}} - RC_0^{\frac{1}{2}} c_{\infty}^{\frac{1}{2}} \quad (xvi)$$

so that from (xv)—

$$\frac{Wl}{V} \cdot \frac{RC_0^{\frac{1}{2}}}{C_0} = \frac{1 - c_{\infty}}{c_{\infty}^{\frac{1}{2}}} \quad (xvii)$$

and finally from (xiv)—

$$X_l = \frac{1 - c_{\infty}}{c_{\infty}^{\frac{1}{2}}} \cdot \frac{KV}{v} \quad (xviii)$$

Thus the length of the package in the new variables is determined by the final exhaustion and the composite factor v/KV involving the rate of liquor flow. This is also the factor multiplying the integral in equation (xii), and so we see that to specify the dyeing behaviour three parameters are needed.

DYEING BEHAVIOUR PREDICTED BY THE MODEL

The equations derived in the last section showed that dyeing behaviour is determined by three parameters—

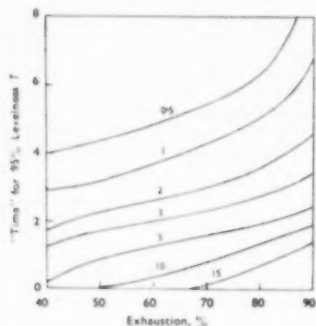
- (1) Final percentage exhaustion of the dyebath
- (2) The constant K , expressing the rate of exchange of dye between liquor and yarn within the package.

- (3) The composite factor v/KV where v is the rate of flow and V the volume of the dyebath.

The dyeing behaviour predicted by the model is presented in the series of graphs shown in Fig. 3-9. These have been obtained by evaluating solutions of the equations for different combinations of the three basic parameters. We are interested mainly in the rate at which final levelness and percentage exhaustion are approached. In order to have a quantitative measure of levelness we define a "levelness factor", which is the ratio of the lowest to the highest concentration of dye on the yarn existing anywhere in the package at the time at which the levelness is examined.

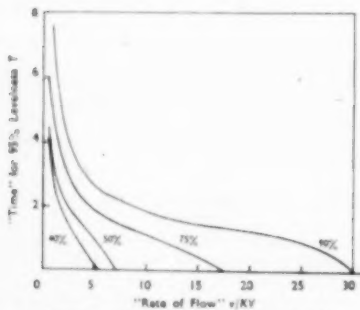
The main conclusions to be drawn from the calculated results are these—

(a) Time to reach levelness for any given flow rate depends on the degree of final exhaustion of the dyebath, being shorter at low percentage exhaustions. The influence of exhaustion is shown in Fig. 3 for a number of different flow rates. The technical features—volume ratio, depth of shade, dye concentration—are all included in the expression for percentage exhaustion. They influence the approach to levelness through their effect on



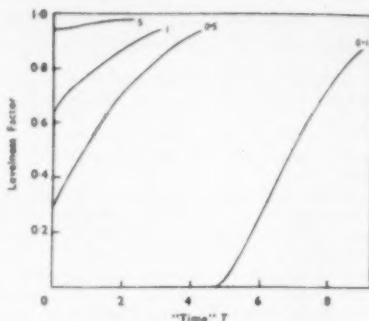
Figures on curves are values of "rate of flow" v/KV
Liquor : yarn ratio 20 : 1

FIG. 3



Figures on curves are final percentage exhaustions
Liquor : yarn ratio 20 : 1

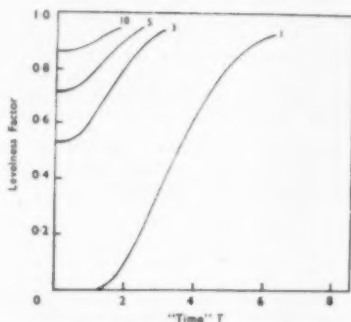
FIG. 4



Figures on curves are values of "rate of flow" v/KV

Liquor : yarn ratio ... 20 : 1
Final exhaustion ... 50%

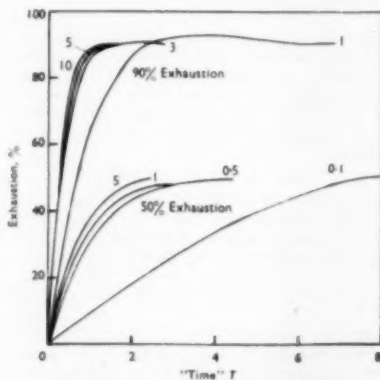
FIG. 5



Figures on curves are values of "rate of flow" v/KV

Liquor : yarn ratio ... 20 : 1
Final exhaustion ... 90%

FIG. 6



Figures on curves are values of "rate of flow" v/KV
Liquor : yarn ratio 20 : 1

FIG. 7

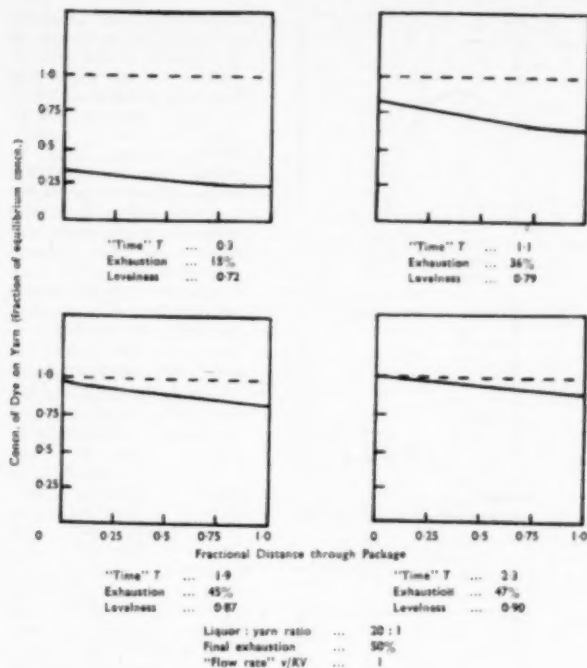


FIG. 8

exhaustion. Salt concentration and temperature will influence exhaustion as well, but their effect on K and the other two parameters must be allowed for in order to estimate their influence on the dyeing process.

(b) Time of levelness for a given final exhaustion decreases with increasing flow rate. The curves of Fig. 4 show that for each given exhaustion there is a flow rate for which levelness is attained almost immediately dyeing commences. Obviously no improvement is possible (as regards levelness) by using higher flow rates. As the flow rate is decreased time to levelness increases, and this increase becomes very marked at very low flow rates.

(c) The way in which the final levelness and equilibrium exhaustion are approached is shown in Fig. 5-7 for final exhaustions of 50% and 90% and several rates of flow. For each exhaustion there is a range of relatively high flow rates for which a high degree of levelness is attained theoretically as soon as liquor flow begins, and a range of lower rates for which the levelness factor is zero in the early stages of dyeing. Comparison of the levelness curves of Fig. 5 and 6 with the exhaustion curves of Fig. 7 shows that for a flow rate given by $v/KV = 5$ and for 50% final exhaustion, for example, levelness is obtained very soon after commencement of dyeing, but the equilibrium exhaustion is

not approached till much later. With 90% final exhaustion and the same rate of flow, or with 50% exhaustion and a lower flow rate, the time to reach levelness exceeds that required to approach equilibrium exhaustion.

(d) The results stated in (c) are to some extent clarified by the curves of Fig. 8 and 9. These show how the dye is distributed through the package at various times for a final exhaustion of 50% in one case and 98% in the other. The latter exhaustion is chosen abnormally high to exaggerate the effects associated with high final exhaustion. At low exhaustions a high degree of levelness is established early in the dyeing process and maintained to equilibrium (Fig. 8). At high exhaustion (Fig. 9) an overdyed region is rapidly established where the dye liquor first makes contact with the package. All the dye originally in the bath is taken up by this overdyed region, and for a long time the circulating dye liquor contains no dye. During this time dye is merely being

washed off an overdyed region and deposited on the neighbouring, less heavily dyed region. Thus the overdyed region gradually moves along the package, its concentration diminishing as it moves. The excess concentration may still be appreciable when the overdyed region reaches the end of the package, in which case some dye is washed off and returns to the dyebath to be deposited on the yarn again where the liquor enters the package. The time required for this sequence of events is long compared with that required to exhaust the bath to a degree corresponding roughly with final equilibrium.

(e) Apart from the fact that the exponent in the absorption isotherm (ii) differs to some extent for different dyes, the characteristics of any particular dye appear in the model only through the rate constant K . There is no point in introducing specific isotherms until further evidence is available as to the correctness or otherwise of the law of exchange of dye between yarn and liquor expressed by equation (i). The results so far discussed are therefore quite general, and as no choice of K has been made they apply, subject to the limitations of the model, to any dye. We shall not consider individual dyes in any detail, except to draw attention to an interesting and important point of difference between dyes for which the local exchange between liquor and yarn is slow

Concn. of Dye on Yarn (fraction of equilibrium concn.)

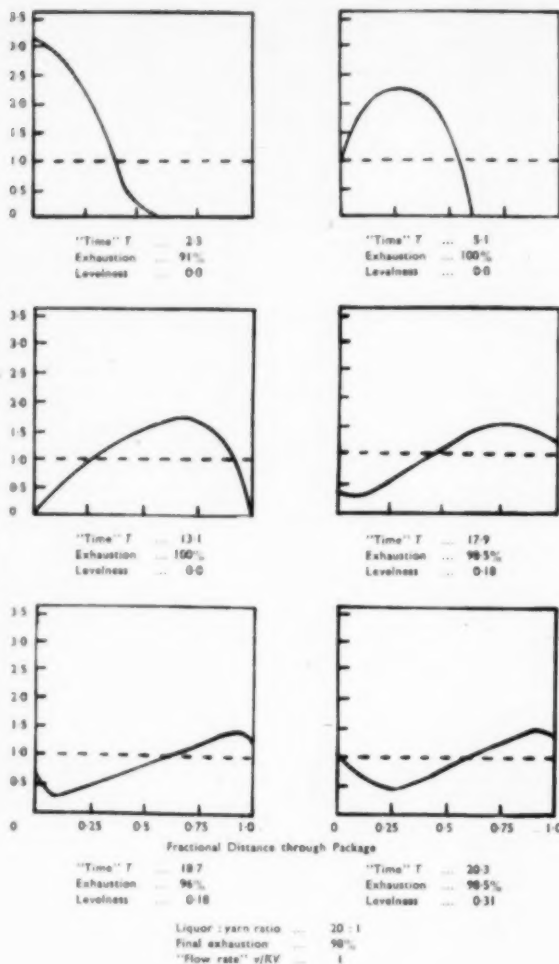


FIG. 9

and those for which it is rapid. These will be described as *slow* and *rapid* dyes, corresponding to small and large values of K respectively. In

TABLE I

Liquor : yarn ratio, ...	20 : 1			
Rate of liquor flow, ...	1 g./min. per g. yarn			
Equilibrium exhaustion, ...	50%			
K , min. ⁻¹ , ...	0.00144	0.0141	0.05	0.10
v/KV , ...	35	3.5	1.0	0.5
Time for 95% levelness*, min., ...	0	91	64	44
Time of half dyeing in package form, min., ...	155	37	12	7

* 95% levelness corresponds to a levelness factor of 0.95

some cases it may be that the speed of this exchange is determined by the rate of diffusion of dye within the filaments, and then a large K and rapid exchange will be associated with a rapidly diffusing (Class A) dye. In other cases, however, the exchange may be primarily a process occurring on the surfaces of the filaments, and diffusion may be relatively unimportant. At the moment we are not concerned with the detailed mechanism but only with the fact that some dyes are taken up slowly and others rapidly.

In Table I are shown the times to reach levelness and the times of half-dyeing in package form for four arbitrary values of K . The point of interest about Table I is that both slow ($K = 0.00144$) and rapid ($K = 0.1$) dyes level more quickly than those of intermediate rates of exchange ($K = 0.0141$ and 0.05). For the slow dyes, however, the time of half-dyeing is long, and this determines dyeing time; whereas for the rapid dyes the time of half-dyeing is short, and the time to level will usually be the deciding factor.

There is this essential difference between the behaviour of the slow and rapid dyes: a slow dye is level almost from the start (as in Fig. 8, for example) and remains level; whereas a rapid dye is unlevel at first (as in Fig. 9), but because the washing off from the overdyed regions and the subsequent take-up on underdyed regions are rapid, the actual time to reach levelness is still reasonably short. This cannot be detected from the values of flow rate or of K alone, but is determined by the parameter v/KV . If v/KV is large, dye is evenly distributed throughout the package from the start; if v/KV is small, uneven distribution occurs at first, and has to be levelled out by subsequent circulation of the dye liquor. Whether this levelling occurs in a short or a long time depends on the value of K .

(f) A further consequence of the appearance of the composite factor v/KV is that the shorter the package the sooner the dyeing level for a given total weight of yarn and given linear flow rate. The relevant factor is the fraction of the total

dyebath content which flows through the package every second. Thus for a given liquor : yarn ratio the linear flow rate required to achieve levelness in a certain time is less for a short package of large cross-section than for a long one of small cross-section.

CONCLUSIONS

The conclusions about package dyeing based on the model are in general agreement with observations from dyeing practice. The model serves to emphasise the necessity of avoiding high exhaustion of the dyebath and confirms that level dyeing is most likely to be secured by rapid flow rate and low exhaustion. For a given exhaustion there is a somewhat critical rate of flow below which the time of levelling increases unduly rapidly. For a given flow rate there is similarly a critical degree of exhaustion above which the time of levelling again increases rapidly. The calculated curves show how the final equilibrium dyeing is approached, and how the dye is distributed through the package at various times. The type of unevenness produced by high exhaustion and/or low flow rate is seen to be associated with the circulation of an almost completely exhausted dye liquor for a considerable time.

We may summarise the information afforded by practical experience and by the theoretical model in the following list of ways in which the dyer may attempt to produce uniformly dyed packages—

1. Avoid any appreciable exhaustion of the dyebath by using a very high concentration of dye, or a large dyebath, or otherwise.

This does not offer a feasible method. Using an infinite concentration of dye or an infinite volume, the dyer could not easily produce light shades and could not readily keep control over exhaustion by such methods as the progressive addition of electrolytes. This method would imply an excessive wastage of dye; it is not possible to use stock dyebaths for controlled direct or vat dyeings in circulatory systems.

2. Arrange for a very slow initial absorption (strike) of the dye, and thereafter control dye substantivity to provide for a steadily and smoothly increasing dyebath exhaustion.

This method is, in fact, already used. In the application of direct dyes¹⁻³ as far as possible salt-free dyebaths are employed, and exhaustion control is maintained by the progressive addition of salt after the strike has taken place and/or by control of dyeing temperature. In the application of vat dyes⁴⁻⁶ the dye is distributed in the non-substantive form as a dispersion of the insoluble oxidised dye, and rendered substantive *in situ*.

3. Use very rapidly diffusing dyes. This does provide an ideal method, but, in practice, rapidly diffusing dyes are those least fast to washing, and incidentally include in the case of cellulosic fibres those which are least fast to light. The dyer must choose his dyes according to fastness for the use in view, and can never depend upon choosing the Class A⁷ rapidly levelling dyes which represent this type.

4. Arrange for high rates of liquor flow, which will compensate for any conceivable rate of exhaustion.

Attention must be given to this aspect in the design of dyeing machinery. To a great extent, however, flow rate depends upon the nature and density of the fibre mass through which dye liquor must flow. The true rate at which liquor can permeate any package will depend upon the resistance offered by the package, and this may or may not be uniform and may be small or great. Much can be done by packing material uniformly in Obermaier-type dyeing machines and in improving the winding of packages of yarn intended for the Longelose type. Wound yarn packages should be made so that maximum stability of package shape alongside maximum openness of structure is achieved. These two attributes are not necessarily mutually opposed, and much might be done by a study of winding speeds, traverse angles, and package geometry to produce the most permeable stable cheeses, cones, etc. By studying these aspects in the case of spun continuous viscose rayon cakes, considerable advances have already been made by the rayon industry. The result has been that all classes of cellulose-substantive dyes can now be applied commercially to viscose yarn in cake form, with consequent advances in dyed yarn quality and avoidance of a winding operation^{2,4}. The need for high flow rates leads to the necessity for employing packages of uniform permeability which are not disturbed under the influence of the high hydrodynamic pressures required. The pressure which any given package will withstand without channelling or deforming is one limiting factor to be accounted for in designing fixed packages such as cakes, cheeses, cones, and tops, or in arranging for the packing down into a container of loose fibre and sliver. Once channelling has taken place, flow rate through the package as a whole is effectively reduced.

5. Reduce the package thickness so that the distance which dye liquor must penetrate, measured in the direction of liquor flow, is at a minimum.

Prepared packages should be as thin-walled as possible, though there is clearly a minimum thickness of penetrable wall below which it is uneconomic to go. There are obvious technical reasons, too, why cones or cheeses should be the shape they are and carry the weight they must. As to box-spun viscose cakes, there is a technical limit below which the thickness of the wall cannot go if the cake is to be a stable unit. What should be noted is that prepared packages should never be thicker than they need be. In the case of a bed, or a wall, of packed loose fibre or sliver or hanks, economic reasons dictate the width of the annular cage space in Obermaier machines, and the thickness of a flat bed in the types of circulatory machines used by loose and slubbing dyers. The thinner the bed, the bigger the machine and the smaller, for a given machine, the production per unit.

6. Continue dyeing for times much longer than those now in use.

This, again, is a matter of economics. It can be taken seriously only in cases where the loss due to faulty dyeing is ultimately greater than the saving of time achieved by short dyeing times. The commercial dyer must decide that for himself. The fact remains that in some sections of the industry the dyeing, for example, of a fine-denier or fine-count fibre or yarn, which beds down densely, is expected to require no longer than a coarse fibre or yarn which gives a permeable package. Longer dyeing times generally increase the difficulty in subsequent processing of the material, and therefore on technical grounds there is a limit to the length of time dyeing should be permitted to proceed.

We might summarise the indications provided by the above model of dyeing as follows—

(a) The dyer must gain still greater control over substantivity and exhaustion rate. In the case of direct and vat dyeing, physicochemical studies have indicated how this might be done, and it is being done at the present time.

(b) Everything possible should be done to increase rate of flow of dye liquor in circulatory

systems through uniformly packed masses, to the extent of the redesigning of winding and packing methods and consideration of machine container shape and means of liquor circulation.

* * *

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The Properties of Some Derivatives of Indanthrone and Flavanthrone

WILLIAM BRADLEY, EDWARD LEETE, and HARRY E. NÜRSTEN

The stability of indanthrone towards oxidation by hypochlorites is enhanced by methylation of the two -NH- groups. The stability is also increased by the introduction of 3-alkylsulphonyl substituents, but not by 3-*tert.*-butyl groups, an indication that the electrical effect of a 3-substituent is more important than its size. *N*-Methylation of indanthrone diminishes its affinity for cellulose, and the same effect is seen on methylating 1,2'- and 2,2'-dianthraquinonylamines. 3,3'-Dibromo-*NN'*-dimethylindanthrone possesses normal affinity. The causes of these effects are considered.

Indanthrone (I), the first vat dye of the anthraquinone series to attain commercial importance, is characterised by several properties. Amongst these are its very sparing solubility in organic solvents, the ease with which it is oxidised to the yellow azine (II), and the marked affinity of its reduced form for cellulose.



The ease of oxidation with alkali-metal hypochlorites leads to difficulties in the application of indanthrone, and methods have been employed or suggested to overcome it. One of the most successful has been the introduction of halogen substituents into appropriate positions in the molecule, as in Caledon Blue RC (III: X = Cl). Another device is to replace the hydrogens of the -NH- groups by alkyl substituents, such as methyl. Indanthren Blue RK (*C.I.* 1108) was stated to be *NN'*-dimethylindanthrone.

NUCLEAR-SUBSTITUTED INDANTHRONES

It is generally known that the introduction of halogen substituents into an aromatic amine diminishes its tendency towards oxidation. The effect is commonly ascribed to the electrical character of the halogens, which bind electrons more strongly than does hydrogen. The effect of halogens in enhancing the stability of indanthrone towards oxidants can be ascribed to the same cause, but there is another possibility. The result may be due, at least in part, to the protection afforded to the -NH- groups by the relatively bulky halogen substituents.

With these points in mind we have prepared¹ two members of the 3,3'-dialkylsulphonylindanthrone series (III: X = Alk-SO₂) in which the alkyls are ethyl and *n*-butyl respectively. The alkylsulphonyl substituent is more bulky and more

strongly electron-attracting than either chlorine or bromine, and it would be expected that these two compounds would be more stable towards oxidation than either indanthrone or its halogenated derivatives. This view proved to be correct. The two alkylsulphonylindanthrones differed from existing dyes in other respects. 3:3'-Diethylsulphonylindanthrone (III: $X = C_2H_5SO_2$) was soluble only with difficulty in alkaline hydrosulphite. The colour of the vat was blue-green, and the affinity for cotton was somewhat weaker than with indanthrone; the final shade was a bright greenish blue. The di-*n*-butylsulphonylindanthrone (III: $X = C_4H_9SO_2$) was reduced with still greater difficulty; the final shade was blue and very weak.

In order to assess the contribution of the size of the alkylsulphonyl groups to the stability towards oxidants we have prepared² 3:3'-di-*tert*-butylindanthrone (III: $X = C(CH_3)_3$). The colouring matter dissolved with difficulty in alkaline hydrosulphite and the affinity for cotton was weak. The final shade was greenish blue. The dyeing proved to be very easily oxidised by hypochlorites. The effect of nuclear alkyl substituents in an aromatic amine is to facilitate oxidation, and account must be taken of this fact in interpreting the result. It is clear, however, that size *per se* is insufficient to enable a nuclear substituent to prevent the oxidation of indanthrone.

N-SUBSTITUTED INDANTHRONES

Indanthren Blue RK, to which reference has been made, was considered by its discoverer to be *NN'*-dimethylindanthrone. Fraser Thomson³ pointed out that the hypochlorite fastness of this colouring matter was much the same as that of indanthrone itself, and one of us suggested⁴ that it was more likely to be the *N*-monomethyl derivative, because of the green coloration which was given very readily when alcoholic potassium hydroxide was added to a solution of the colouring matter in pyridine. We have now prepared⁵ both *N*-methylindanthrone and *NN'*-dimethylindanthrone. The dimethyl compound is quite different from Indanthren Blue RK. It is blue in colour, shows no colour change with alkali in pyridine, and is quite unaffected by sodium hypochlorite. The last observation makes it clear that replacing the hydrogen of -NH- groups by methyl is effective in conferring hypochlorite stability on indanthrone. At the same time the affinity for cellulose is almost completely lost. The monomethyl compound gives a green coloration with alkali in pyridine; its affinity for cellulose lies between that of indanthrone and its *NN'*-dimethyl derivative. It closely resembles Indanthren Blue RK.

In many instances the introduction of halogen substituents increases the affinity of vat dyes for cellulose, and with this in mind we have prepared the 3:3'-dibromo derivatives of *N*-methyl- and *NN'*-dimethyl-indanthrone. 3:3'-Dibromo-*N*-methylindanthrone is readily soluble in alkaline hydrosulphite forming an orange-brown vat; the affinity for cellulose approximates to that of 3:3'-dibromoindanthrone (III: $X = Br$). The

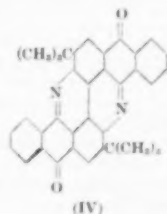
shade is blue, becoming slightly paler on treatment with sodium hypochlorite. 3:3'-Dibromo-*NN'*-dimethylindanthrone⁶ is also readily soluble in alkaline hydrosulphite; it forms an orange-red vat, and from this cellulose is dyed strongly a violet-blue which undergoes no change with sodium hypochlorite.

In this instance, as with the indigoid dyes, the introduction of bromine substituents increases the affinity; at the same time 3:3'-dibromo-*NN'*-dimethylindanthrone provides a further example of the stability towards hypochlorite which results from the methylation of -NH- groups.

General Considerations

1. SOLUBILITY

Whilst indanthrone is very sparingly soluble in organic solvents, its *N*-methyl derivative is more soluble, and its *NN'*-dimethyl derivative much more soluble. A similar relation is found between the solubilities of 3:3'-dibromoindanthrone and its *N*-methyl derivatives. The effect of *N*-methylation is understandable if intramolecular bonding occurs in indanthrone between the -CO- and -NH- groups of several molecules, such as has been suggested for the fibrous proteins⁷. The same explanation would account for the ready solubility of tetra-acetyl-*NN'*-dihydroanthraquinoneazine by Scholl and Berblinger⁸. Hydrogen bonding is probably responsible also for the sparing solubility of 1:2'-dianthraquinonylamine (*C.I.* 1137) and the isomeric 2:2'-compound. In each instance the replacement of the hydrogen of the -NH- group by methyl increases the solubility in organic solvents considerably. We have recently described⁹ an isomer of indanthrone which, though containing -NH- groups, is incapable of the same degree of hydrogen bonding as indanthrone; it is also more soluble than indanthrone. Finally, the two dialkylsulphonylindanthrones, in which the bulky substituents must hinder close packing of the molecules, are both more soluble than indanthrone. Apart from their effect in suppressing hydrogen bonding, it does appear, however, that alkyl groups may have a specific effect in promoting the solubility of vat dyes in organic solvents. Thus we have found² that, whilst flavanthrone is exceedingly sparingly soluble, a di-*tert*-butyl derivative (IV) of this colouring matter is quite readily soluble.



2. VATTING AND AFFINITY

The importance for affinity for cellulose of coplanarity in the molecules of vat dyes and direct

dyes has been stressed by Hodgson and his co-workers¹⁰, and by Clibbens¹¹, who has referred to the weak affinity of Indanthren Blue RK. The possibility of hydrogen bonding between dye and cellulose is probably important also¹². We have found, for instance, that both 1:2' and 2:2'-dianthraquinonylamine have considerable affinity for cellulose, whilst their *N*-methyl derivatives are taken up to a much smaller degree. We have now encountered an instance in di-*tert*-butylflavanthronone (IV), however, in which the parent dye flavanthronone is planar, and the derivative (IV) non-planar. Neither dye contains an -NH- group, and both have good affinity for cellulose. Finally, the molecule of 3,3'-dibromo-*NN'*-dimethylindanthronone cannot be planar, yet the compound has good affinity for cellulose.

DYEINGS

In order to obtain 3% dyeings, the dye (0.03 g.) was dissolved in concentrated sulphuric acid (3 c.c.) by stirring for 15 min. The solution was poured into water (40 c.c.) in such a manner as to keep the temperature below 40°C. The suspension was centrifuged and decanted, and the finely divided precipitate washed with water (25 c.c.), and then with 5% aqueous sodium hydroxide (25 c.c.) by centrifuging and decanting. The dye was vatted for 15 min. at 50-60°C. with sodium hydrosulphite (0.1 g.) and 5% aqueous sodium hydroxide (3 c.c.) and, after warm water (25 c.c.) had been added, dyed on cotton (1 g.) for 45 min. at the same temperature, more sodium hydrosulphite being added if necessary. An exhaust dyeing was obtained by dyeing fresh cotton (1 g.) for a further 30 min. After souring, the dyeing was soaped in a solution containing 2 g. soap per litre by raising to the boil and allowing to cool. The above procedure closely imitates Method I accepted

by the Vat Dyes Committee¹³. Distilled water was used throughout.

FASTNESS TO WASHING

The dyeing was boiled for 30 min. in a liquor (50 c.c.) containing soap (5 g./litre) and sodium carbonate (2 g./litre). This test was based on Test 3 prescribed by the Society¹⁴. Dimethylindanthronone showed appreciable loss in the washing test. The remainder of the compounds described showed little change.

* * *

The results described in the above paper were obtained during the tenure of a Bradford Dyers' Association Research Fellowship (H.E.N.), and of a Clothworkers Research Scholarship and a grant from Messrs. Courtaulds' Scientific and Educational Trust Fund (E.L.). We desire to express our grateful thanks to the donors of these awards.

CLOTHWORKERS RESEARCH LABORATORY
UNIVERSITY OF LEEDS

(Received 16th November 1951)

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Notes

Proceedings of the Council

At a meeting of the Council held at the Victoria Hotel, Bradford, on 13th February 1952, the proceedings included the following items of interest—

FASTNESS AND WEARABILITY.—A suggestion of Dr. C. M. Whittaker, that a symposium should be held on these subjects, was recorded for future consideration.

NEW BYE-LAWS.—Revised drafts of Bye-laws 12, 16, 22, and 24 were accepted for presentation to the A.G.M. It was resolved that Council's gratitude be conveyed to the members of the Bye-laws Committee.

AIRMAIL BALLOT PAPERS.—A recommendation of the Finance Committee, that ballot papers to members resident outside Europe be sent by airmail, was accepted.

MEMBERSHIP.—Twenty-four applications for ordinary membership and three for junior membership were approved. Mr. W. J. B. Lee was readmitted to membership.

Meetings of Council and Committees

March

Council—12th
Finance—12th
Publications—18th
Terms and Definitions—7th
Summer School—8th
Colour Index Editorial Panel—14th
Fastness Tests Co-ordinating—25th
Printing Symposium—25th

Deaths

We regret to report the loss by death of Mr. G. D. Barrow, Mr. O. Boothby, Mr. C. S. Groves, Mr. J. R. Hogg, and Mr. N. Holmberg.

Appointment of Indexer

The Society is very grateful to Mrs. E. G. Thomson for undertaking the indexing of the *Journal*. As a result of her efforts it has been possible to publish the Index to volume 67 with the February 1952 issue.

V.T.C.C. Annual Conference Bad Neuenahr, 1st-4th May 1952

The programme includes papers from England, France, and Switzerland as well as Germany. Full particulars may be obtained from, and accommodation booked through, the Verein der Textil-Chemiker und Coloristen, Staatliches Institut für Textilchemie, (17b) Badenweiler, Germany.

The Association of Heavy Textile Prooferers of Great Britain

For some years the firms throughout the country engaged in the proofing of heavy fabrics made from flax, cotton, hemp, and jute have felt the need for such an organisation, the primary object of which will be the providing of a service to the users of all types of proofed canvasses. The above association was therefore formed on 15th November 1951, and begins with a membership representing all the firms engaged in the industry. A Technical Committee has been appointed to deal with all matters of a scientific and technical nature. The Secretaries are Messrs. Davidson, Downie & McGown, C.A., of 135 Buchanan Street, Glasgow C.1.

New American Rules for Use of the Terms Rayon and Acetate

Under new trade practice rules issued on 11th Dec. 1951 by the U.S. Federal Trade Commission, the following terms and definitions are obligatory in the U.S.A.—*Rayon*: "man-made textile fibers and filaments composed of regenerated cellulose"; and *Acetate*: "man-made textile fibers and filaments composed of cellulose acetate".

c o c

Patent Office Library—Opening Hours

The extended hours of opening, viz. Monday to Friday 10 a.m.-9 p.m. and Saturday 10 a.m.-5 p.m., will continue in force until further notice.

c o c

Algil—Polystyrene Fibre

A new polystyrene fibre of unusual fineness is now being produced in the U.S.A. by Polymers Inc. under the name of Algil.

c o c

Misrophan—an Egyptian Glass Fibre

Misrophan is a glass fibre made by the Société Égyptienne Misr pour la Rayonne in their works at Kafr el Dawar near Alexandria. Production in 1950 was 187 tons.

c o c

Centenary of the Founding of Bottomley & Emerson Ltd. Brighouse and Huddersfield

The firm of Bottomley & Emerson Ltd. was founded in 1841, when John Carr Bottomley started business in Brighouse as a drysalter, some of his descendants being among the company's directors. The firm continued as drysalters and chemical manufacturers until 1919, when they amalgamated with the firm of Dawson & Emerson of Huddersfield, who started in business in 1912 as chemical manufacturers and dye merchants. It was then that the company commenced to make azo dyes, and later stilbene and triphenylmethane dyes, in which work it is still engaged as an independent concern. A fuller account of this firm's history is given in the *Dyer*, 106, 891-892 (28th Dec. 1951).

c o c

OBITUARY NOTICES

James Baddiley

A PERSONAL APPRECIATION

James Baddiley and I were students together in the Department of Colour Chemistry at Leeds University under Professor A. G. Green forty years ago. Horsfall and Haley, Yeoman and Davies, all of whom touched the industry beneficently in later years, were there at the same time. F. Scholefield had just preceded us; F. M. Rowe came soon after. It need not be said that any of us dazzled our contemporaries, and if ours was later described as a vintage year, it was not that there was great virtue in the vines; it was the soil that was kindly and the elements that favoured us, for we finished our academic training in time for the best of us to help, in the hasty unnatural expansion of the British dyestuff industry which began in 1914. So if Baddiley stood out as being a little different from the rest of us, and he did, it was not for originality of mind nor for prodigious industry nor for any passion for knowledge, but because, in his quiet way, he knew more about the art of living than most of us. Certainly there were then no signs of the great ability latent in him.

His family farmed in South Yorkshire, and he

came to Leeds, if I remember aright, from Bradford Grammar School as a chemistry student. He was tall, fair of complexion, and stooped a little. He had a benign but not a jovial air, and was, from self-respect and not at all from vanity, almost an exquisite in his dress. His brown shoes had a deeper, richer lustre and a less yellow tone than ours, and at a time when the waistcoat was still a means of adornment, his always had a sedate elegance. His shirts, I remember, were usually a cream-coloured silk because, so he said, they lasted longer and were in the long run cheaper. He never hurried because he always had plenty of time. I never saw him in a rage nor ever heard him speak bitterly of anybody. Violence of speech, thought, or action he abhorred. He worked steadily and well but in moderation, and the laboratory did not keep him from a *matinée* at the local music hall if the bill promised very well. He read the *New Age* in the days when it was edited by Orage, and also took another weekly run by Belloc and the two Chestertons of which I have forgotten the name. He had a fondness for chamber music, and we went together to the infrequent concerts given at the Hotel Metropole. At that time, too, Constance Garnett's translations of the Russian novelists

were being issued. Turgenev he liked, Tolstoy he admired, Dostoevsky he detested for a hysterical and disorderly mind. He always seemed to have more money than he needed, was generous without ostentation, and in the long vacation often went to Germany—not to study but to enjoy himself. I hope from this that you have a picture of him in his early twenties. Gentler than most, kinder than most, more modest, reserved and fastidious than most of us, he was a civilised man with wide interests. Do I remember any incident of those days which describes him? Of many, this one must suffice. In my last year at Leeds I was assistant to Professor Green in his consulting practice. On leaving him to take up my first appointment in industry, Green asked me whom I would recommend as my successor. I said "Without any doubt Baddiley; he has, so to speak, got the hang of chemistry." "You are letting your friendship mislead you", said Green coldly, "he doesn't take the subject seriously."

When from the University he went to work in Levinsteins' laboratories in Blackley, chance also took me to Manchester, and our earlier friendship continued and deepened. There his social interests broadened still more. Painting now interested him, Rowley Smart and Henry Vitofski were his friends, he even bought their pictures. He shared rooms with a man on the staff of the *Manchester Guardian* and through him became friends with Stanley Houghton, who wrote *Hindle Wakes*, and with Maltby, another dramatist of the time. Usually he went at least once a week to Miss Horniman's Gaiety Theatre, then, beyond question, the most intelligent theatre in England. At that time, too, he was a member of two or three clubs in the Rusholme district which were frequented mainly by writers and newspaper men. These places only came to life well after midnight, after the restaurants at the Midland Hotel were closed and the newspapers put to bed. There Baddiley would gossip with kindness, or argue with wit, till dawn on any subjects but money and work. The man was the student, ripened. Indeed the only marked change in him, and the atmosphere at Blackley was probably responsible for it, was a fondness for going each week-end to the wilder, bleaker hills of Derbyshire.

Later the circumstances of the times separated us, and it was not till the nineteen-twenties, when I went to live in Lancashire, that our friendship was renewed with the old intimacy. By this time we were both married and had children of the same ages, so Baddiley often brought his wife and children to my house and we frequented his home at Fallowfield, the most hospitable house I have ever known. I think these must have been his happiest years. He had young children and a devoted wife, he was doing well at his work, and if he had responsibility there, it did not worry him. He had plenty of friends, and on at least one evening in every week the house was full of them. We had supper in relays, first of all the children—there usually were, or seemed to be, about a dozen—then, when the children were in bed or sent home, the men of the party sat down together, and

last of all the women, who had been waiting on us all. Baddiley was happily busy all the time, now dealing gently with unreasonably exuberant children, now opening the bottles or carving the birds, and, after seeing that the womenfolk didn't suffer by waiting, would tell us that half-past one o'clock in the morning wasn't really late.

It was in the early thirties that a break in the curve of his life began to show. In his leisure hours this most sociable of men began to make his home a retreat which he left only with reluctance, though needless to say his friends were as welcome there as ever. He lost interest in the theatre and the concert hall, and the streets knew him no more at midnight. Gradually, too, after his day's work he withdrew himself even from the society of his colleagues. In consequence, since the amenities of friendship call for both going and coming and cannot be carried for long by one-way traffic, his life became sharply divided into two separate phases. There was the day of active, close, and cordial relations with his colleagues and with such of his friends as called to see him at Blackley, and an evening at home entirely isolated from them. Later, after his children were all married and his own and his wife's health became precarious, this self-isolation became almost rigid. His friends were, I think, more welcome than ever, when they visited his home, but he could not be persuaded to leave it for a single evening. At heart he was still the same man, but somehow the direction of his life had changed.

I mention all this with reluctance, but it should be said for the sake of his professional reputation. It is unwise for a chemist to disregard extramural activities. Unfortunately, his standing in the profession depends much on the extent to which he publicly identifies himself with it and with his own work. Baddiley, however, had clearly decided that his official responsibilities as Director of Research for I.C.I. at Blackley finished at six o'clock in the evening, and in consequence his memorable directorship of a research organisation of outstanding national value was for many years cloaked in anonymity. I said this to him shortly before he retired from his directorship. This was the substance of his reply: "Money for money's sake has never interested me. I count myself a very fortunate man. I have had a great opportunity and I think I have been equal to it. There is no other job I could have done so well, no other work I should have liked so much. What more could a man ask for?" "What I should have liked", I answered, "is more awareness of you in the academic and chemical world." "That does not matter", he replied, "at Blackley, I have had colleagues and a chairman who believed in me, supported me loyally in difficult times, and found all the money the department needed. That is what I call real recognition."

Well, that is the way he would have it, and the choice was his to make. P. F. CHOSLAND

AN APPRECIATION OF HIS WORK

In the death of James Baddiley, on 26th September 1951, the Manchester Section of the

Society lost one of its oldest members and one whose great services in the renaissance of the dyestuffs industry in Great Britain, in the first world war and subsequently, had been recognised by the award to him, in February 1939, of the Society's Perkin Medal. Born in Yorkshire on 15th July 1885, Baddiley was educated at Bradford Grammar School and Leeds University, where he graduated with first-class honours in 1907. For the next two years he worked with Professor A. G. Green in the Clothworkers Research Laboratory, chiefly on the colouring matters of the stilbene group. In this



investigation Baddiley and Green found that those derivatives of *p*-nitrotoluene containing in the *ortho* position an electropositive or weakly electronegative group, which react least readily with caustic alkalis, could also be converted into dinitrostilbene derivatives by carrying the combined condensation and oxidation further. They also showed that 2,4-dinitrotoluene is far more reactive, and with cold alcoholic potash and iodine was converted into 2,2':4,4'-tetranitrostilbene without intermediate formation of the dibenzyl compound. The paper¹ which describes this investigation is the only scientific paper which stands in Baddiley's name, although his name appears as co-inventor on some 60 British patent specifications.

In 1909 he came to Blackley as research chemist with Levinstein Ltd., and during the next five years, with Dr. H. Levinstein, who had just taken over the management of the works, he took out twelve British patents relating to azo dyes and intermediates. Seven of these patents² related to the fixation of azo dyes on vegetable fibres by after-

treatment with formaldehyde or the preparation of azo dyes suitable for treatment in this manner. Dyes of this type were being marketed by Levinstein Ltd. at the time as their well known Vulcan range, and Vulcan Black G was the best black then known for dyeing velvet. The remaining patents of this period³ deal with the preparation of derivatives of aminonaphtholsulphonic acids, such as J acid, H acid, or γ acid, suitable for coupling to give azo dyes, or with the preparation of azo dyes from intermediates of this general type, including an alternative to Beyer's Benzo Fast Red 8BL, and an outcome of the work was the marketing of a range of direct cotton dyes fast to light.

This and other work had firmly established Baddiley's position in the industry by the outbreak of war in 1914, and he became Head of the Technical Research Department at Blackley, a position he retained when the British Dyestuffs Corporation was formed by the merging of Levinstein Ltd. and British Dyes Ltd. after the war. Later, after Professor Green had resigned his post as Director of Research, Baddiley became Manager of the Research Department, and in 1930, after British Dyestuffs Corporation had become part of Imperial Chemical Industries Ltd., a delegate director and Joint Technical Manager of the Dyestuffs Group, remaining Director of Research until his retirement in 1947.

The post-war British specifications on which Baddiley's name appears as co-inventor cover a wide field, including not only new dyes of the azo, anthraquinone, and triphenylmethane series, but also the preparation of dispersing or wetting agents, pigments, and intermediates. The most important of his contributions to technical development in this way are, first, his discovery, in 1922, with the late A. Shepherdson, of the valuable dyeing properties of dispersions of unsulphonated amino derivatives of anthraquinone for acetate rayon⁴; and, second, his contribution to the use of dispersions of azo dyes for the same purpose⁵. Both discoveries materially contributed to the development of the use of acetate rayon as a textile fibre, and they are mentioned as two out of five outstanding developments in dye chemistry in the report of the Board of Trade Dyestuffs Development Committee in August 1930. A third discovery, also mentioned by this committee in the report, was that of a special range of azo dyes giving level dyeings on the "irregular" viscose rayon which at that time was frequently encountered⁶.

Although the paper in the *Transactions of the Chemical Society*¹ and these British patents are Baddiley's only contribution to the technical literature of the industry, the review of the development of the industry which he gave when presented with the Perkin Medal is a balanced and authoritative survey of the renaissance of the British industry which will not soon be superseded⁷. With his exceptional knowledge of his subject he combined an ability for clear exposition which found expression notably in this address, in the article on dyes contributed to the 1950 edition of *Chambers's Encyclopedia*, in the surveys which he contributed

to the *Review of Textile Progress*, and in the obituary notice of A. G. Green which he wrote for the Royal Society². The notice last mentioned displays, moreover, not only something of Baddiley's personal loyalties and insight into character, but also his keen sense of historical perspective, and his last contribution, read at the Society's Conference at Harrogate last September during his fatal illness, gave a historical survey of the rise and development of the wool dyes³. In that paper Baddiley made once more the point about the close relations between science and technology, and the way in which the dyestuffs industry is based on organic chemical research, which he had made in his Perkin Medal address. With the introductory remarks to the discussion at Harrogate, this paper shows how to the end he remained a close student of the progress of the dyestuffs and organic chemical industries and retained his ability rapidly to discern the significant trends. They reflect, faithfully, the outlook which was most characteristic of him and views with which his colleagues, whose affection and respect he had long since won, were well familiar. Like the paper "Industry's Debt to Science" which he contributed to the *Journal* in connection with the centenary celebrations of the Chemical Society in July 1947¹⁰, they are a fitting swan song, and summarise much which his colleagues found most stimulating and for which they will long remember him. His view on the key position of the dyestuffs industry and its contribution to organic chemical industry generally, his belief in the value of research and his unwavering support of both research and teaching of the highest standards at the universities, his stress on the necessity for invention rather than imitation, on the importance of welding the scientific and the economic outlooks in the conduct of research, and for the industry to keep in the closest possible touch, not only with the steady advance of ideas and techniques in organic chemistry but also with the technology of the application of dyes and the problems of the user, expressed in these papers, were firmly held and consistently and cogently argued. They may well stand as a fitting memorial to one who, on the research side, had played a decisive part in the renaissance of the British dyestuffs industry.

R. BRIGHTMAN

¹ *Trans. Chem. Soc.*, 1721 (1908).

² *RP* 27,525 (1912), 28,296 (1912), 1435-6 (1913), 25,547 (1913), 28,569 (1913), 8569 (1914).

³ *RP* 11,877 (1910), 15,008 (1910), 15,070 (1910), 26,577 (1910), 12,281 (1911).

⁴ *RP* 211,720.

⁵ *RP* 224,077.

⁶ *CF. RP* 270,446, 287,214, 287,609, etc.

⁷ *J.S.D.C.*, 55, 236-246 (1939).

⁸ *Obituary Notices of Fellows of the Royal Society*, 4 (Nov. 1943); *J.C.S.*, 842 (1946).

⁹ *J.S.D.C.*, 67, 546 (Dec. 1951).

¹⁰ *Ibid.*, 63, 206 (1947).

Fred Ashworth

Fred Ashworth died at the age of fifty-one years, after a very short illness. He was educated at the Royal Technical College, Salford, and also took his

diploma in dyeing with the City and Guilds of London Institute. He was at first employed at the Clayton Works of British Dyestuffs Corporation, and left them to join J. Chadwick & Co. Ltd. of Oldham, where he remained for twenty-four years, latterly being the chief chemist.

Fred Ashworth saw service in the Royal Flying Corps during the 1914-18 war, and in the last war was a hard worker in the Air Training Corps. He was modest and unassuming, with a quiet, but solid interest in the Society. He was an accomplished amateur artist.

He is survived by a widow, a married son, and a married daughter.

J. W. REIDY

William McCallum

We deeply regret to record the death on 26th October 1951 of Mr. W. McCallum of Bradford, a member of this Society for over fifteen years.

He spent the whole of his business life in the service of the British Cotton & Wool Dyers' Association Ltd. Starting in 1900 with Alexander Reid & Brother Ltd., he rose to be the manager of that branch. In 1930 he left Paisley, his birthplace, to take up the position of manager of Heppenstall Bros. Ltd. of Huddersfield. Two years later he became joint manager of William North & Co. Ltd., Crossley Hall Dyeworks, Bradford, a position he held at the time of his death.

All his life Mr. McCallum was deeply interested in social work. He was for a time Chairman of the Paisley Literary Society; he ran the swimming and football sections of the Y.M.C.A.; he had charge of the Coats Memorial Church Scouts, and for a period was treasurer of the Paisley Choral Union. Both in Huddersfield and in Bradford he retained a lively interest in Y.M.C.A. work and was a keen supporter of local football.

For some years prior to his death he had been a member of the Executive Committee of the Bradford Textile Society, of which body he was Chairman during the session 1949-1950.

Willie McCallum was a genial Scot—an excellent mixer—who will be greatly missed by a large number of friends, both inside and outside his business circle. To his widow we extend our deepest sympathy.

E. ISLES

Edgar Thornton

It is with deepest regret that we record the death of Mr. Edgar Thornton, aged seventy-four.

His career commenced as apprentice dyer under the late Mr. H. H. Seddon, and later he became Head Dyer for Edwin Brook & Co. Ltd. After the death of Mr. Edwin Brook he took over the business, and was very successful as one of the best piece dyers in the trade.

Few men have been better liked than Edgar Thornton; he was one of Nature's gentlemen, kind and unassuming, and had a lifelong connection with Honley High Street Methodist Chapel, where he was in the choir.

He leaves a widow and a son, who is carrying on the business.

G. K. SEDDON

New Books and Publications

Review of Textile Progress

Volume II 1950

Compiled and published jointly by the Textile Institute and the Society of Dyers and Colourists. Pp. 482. Price, 25s. 6d.

The second volume of this important annual review is larger than the first, mainly owing to the inclusion of three new sections, viz. "Nylon—Production", by G. Lonsby and A. R. Munden, "Cotton and Rayon—Preparation and Weaving", by E. Cotterill, and "Wool—Preparation and Weaving", by D. C. Snowden. On the other hand, the sections on "Wool Production" and "Textile Microscopy with special reference to the Microscopy of Natural Fibres" do not appear in the current volume. In the next volume it is hoped to include a contribution on "Sizing".

The high standard of the first volume has been fully maintained, and the review continues to reflect great credit on all concerned with its production. It is essential that the joint Committee should not allow the sharp increase in printing costs to hamper their work, which has now established itself as an important contribution to progress in the production and coloration of textile materials.

C. L. BIRD

Identificación de Materias Colorantes sobre Fibras Textiles

y Determinación de los Iones Metálicos en Materiales Fibrosos, Colorantes y Pigmentos Orgánicos

By E. Clayton, translated by C. Mas Gibert and J. Alsina Giralt. Barcelona: Manuel Marin, 1951.

The above is the title of the Spanish version of the Society's publication *Identification of Dyes on Textile Fibres*.

The translation has been carried out word for word where this has been possible, so that the style of the original has been preserved. It is to be regretted that, in an authoritative work such as this, more care was not taken to prevent the many errors encountered. The type of error gives rise to the impression that the translation was not revised.

It is noticed that the translators have a preference for "triphenylmethane" instead of *triarylmethane*, and have used the former exclusively throughout the book.

The efficiency of the translation of technical terms and chemical names is of a high order.

Some errors have been repeated several times, e.g. in Tables VIII, IX, XI, and XII, *weakly acidulated solution* reads "strongly acidulated solution". In the note at the bottom of Table XII, Chrome Green is described as being a diphenylmethane dye. On p. 44 one reads: "*m*-dinitrobenzene test for Sn^{++} ions" instead of *cacotheline* test for Sn^{++} ions, i.e. dinitrobrucine. Again, on p. 20, in the test for sulphur dyes, we are told to note the presence of lead or mercuric "sulphate" on the test paper.

There are other errors, but in general the translation has been well done, and the book should be well received, especially in the Spanish-speaking Latin American countries, where authoritative works of this type are difficult to obtain.

N. PHILLIPS

The Chemical Technology of Dyeing and Printing

Volume II

Substantive, Basic, Acid and Pigment Colours, Aniline Black, and Dyestuffs for Acetate Rayon and Synthetic Fibres

By Louis Diserens, translated and revised from the second German edition by P. Wengraf and H. P. Baumann. Pp. xii + 446. New York: Reinhold Publishing Corp. 1951. Price, \$12.00.

This volume is largely a translation of Volume II of Dr. Diserens's book (reviewed in J.S.D.C., 65, 362 (1949)) together with the portion of Volume III (reviewed in J.S.D.C., 66, 289 (1950)) dealing with pigment printing. The size of the volume has been kept down by omitting chapters dealing with solvents, auxiliary products, and thickening agents, because such information is already available to the English reader.

The present work deals with the application of substantive, basic, acid, and pigment dyes, as well as of Aniline Black. It includes notes on the dyeing and printing of cellulose acetate, nylon, and the other synthetic fibres including glass, special printing effects being dealt with in a separate chapter.

The whole work has been rewritten and is exceptionally well referenced, British literature in particular having been well combed.

These two volumes together cover the printing of all types of dyes on to nearly every variety of textile material, and present to the reader the important developments in recent years happily blended with the wide experience and knowledge of the practical printer.

A. THOMSON

Neueste Fortschritte und Verfahren in der chemischen Technologie der Textilfasern

Erster Teil

Die neuesten Fortschritte in der Anwendung der Farbstoffe

By Louis Diserens. Vol. I, pp. xv + 919. Basle: Verlag Birkhäuser. 3rd edition, revised and expanded. 1951. Price, linen, 122.70 Swiss francs.

The second edition of this book, published in German in 1946 (reviewed in J.S.D.C., 63, 29 (1947)), has been rewritten and greatly expanded (from 653 to 919 pages) by the inclusion of information on new fibres, dyes, auxiliary compounds, and processes.

The same general scheme has been adopted, but the text has been rewritten and brought up to date to January 1950, and includes, in particular, much information on British and American processes,

products, and developments not previously available to the author on account of the war.

The book has, in contrast to the second edition, been carefully written, revised, and edited, and, in view of the shortage of books on printing, this third edition should become one of the standard works of reference, and be welcomed both by the practical printer and by the student.

A. THOMSON

Progrès réalisés dans le domaine de la Technologie chimique des Textiles

Première Partie

Progrès réalisés dans l'Application des Matières colorantes

By LAUREN DISENENS. Vol. I, pp. xii + 573. Paris: Editions Teintex. 2nd, revised edition. 1950. Price, fr. 4200 (France), fr. 4500 (abroad).

Readers of this *Journal* have now become familiar with the excellent series of books on recent progress in dyeing and printing which became available after the war, appearing in German (reviewed in J.S.D.C., 63, 29 (1947); 65, 362 (1949); 66, 289 (1950)), and later translated into English by P. Wengraf and H. P. Baumann (reviewed in J.S.D.C., 65, 235 (1949) and above).

The first French edition of this appeared during 1938-1939, and now a second French edition is to consist of four volumes instead of three. This particular volume is a translation of the first three chapters of the second Swiss edition, and deals with the developments in vat, sulphur, and solubilised vat dyes and in modern methods of their application in dyeing and printing, the information being revised to January 1950.

Additional French patents and references are quoted, and a useful innovation is the inclusion in the tables of auxiliaries of references to both the text of this volume and the literature.

Neither the paper nor the printing is as good as that used for the Swiss edition, but readers who can manage to read French but not German will welcome this new series of books. A. THOMSON

Textile Laboratory Manual

By W. Garner. Pp. x + 574. London: National Trade Press Ltd. 2nd edition 1951. Price, cloth, 30s. 6d.

The new edition follows closely the lines of the first edition of 1949, but has been amplified by some 92 additional pages. The author states in his preface that "because of the desirability of keeping original pages unchanged as far as possible, many minor improvements have had to be omitted". Thus some of the criticisms of the first edition (J.S.D.C., 66, 56 (1950)) remain.

We are glad to note that the methods of the Society's Committee on the Dyeing Properties of Wool Dyes are given, although briefly. We would, however, have expected to find the methods and classification recommended by the Cotton Dyes

Committee, since these have been widely adopted by both dye manufacturers and dye users. Methods of test for vat dyes have also been given by the Society's Vat Dyes Committee. The fastness tests sponsored by the Society are given more fully than in the first edition.

The manual has clearly found its mark, since a second edition is required in the space of two years, and its use in textile laboratories is now fairly general.

P. W. CUNLIFFE

Official Methods of Analysis of the Society of Leather Trades Chemists

Croydon: Society of Leather Trades Chemists. 2nd edition 1951. Pp. vii + 199. Price, cloth, 17s. 6d.

The International Society of Leather Trades Chemists really dates back to 1897. One of its objects was "to establish standard international methods for the valuation of all materials used in the leather industries, especially as regards the drawing of samples from bulk, the processes of analysis employed, and the form in which their results are to be stated". At the dissolution of the I.S.L.T.C. in 1947 the present Society of Leather Trades Chemists took over the existing official methods of analysis, and adopted them as their official methods with one minor exception.

The present volume contains these methods with the modifications and additions which have since been approved by the S.L.T.C. It may be mentioned that the procedure employed in evolving an official method is to form a committee composed of appropriate chemists. A provisional official method is then approved for examination by the chemists in the industry. After a period of two years it is adopted as an official method if approved at a Conference of the S.L.T.C.

Methods are given for the analysis of tanning materials, the pH and copper contents of tanning extracts, vegetable-tanned leather including its acidity, chrome liquors, chrome leather, lime liquors, sodium sulphide, and lactic acid. The methods given for oils and fats are sound, but British Standard 684:1950 has recently been adopted by the S.L.T.C. Methods are also given for sulphated oils, and further methods for determining the -COOH, -OSO₃H, and -SO₃H groups and their salts are also being studied (J.I.S.L.T.C., 30, 306 (1946); 31, 100 (1947)).

One of the most modern parts of the volume is the one dealing with physical methods for the testing of leather.

A copy of the book is essential to any chemist who wishes to analyse tanning materials or leather, because analyses made by any other methods will give results which are not in accordance with those accepted by leather trades chemists and the industry. The methods are under constant observation by the Analytical Committees of the S.L.T.C., and blank pages are provided for any future alterations.

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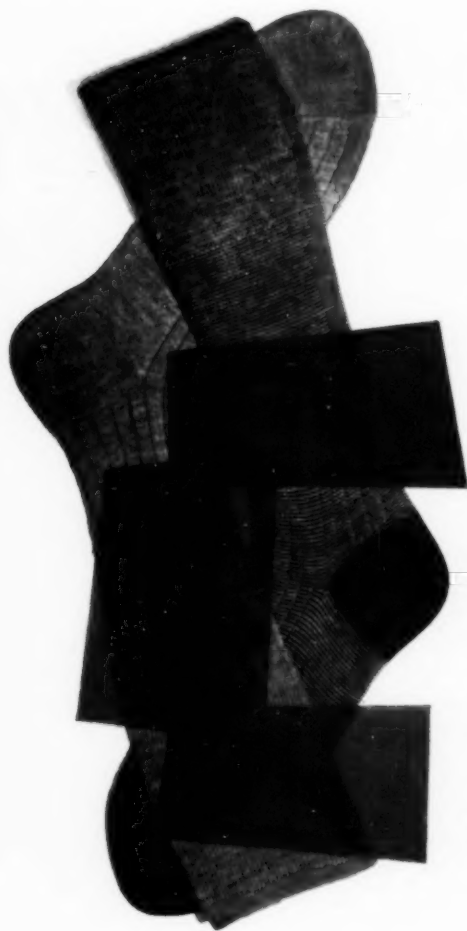
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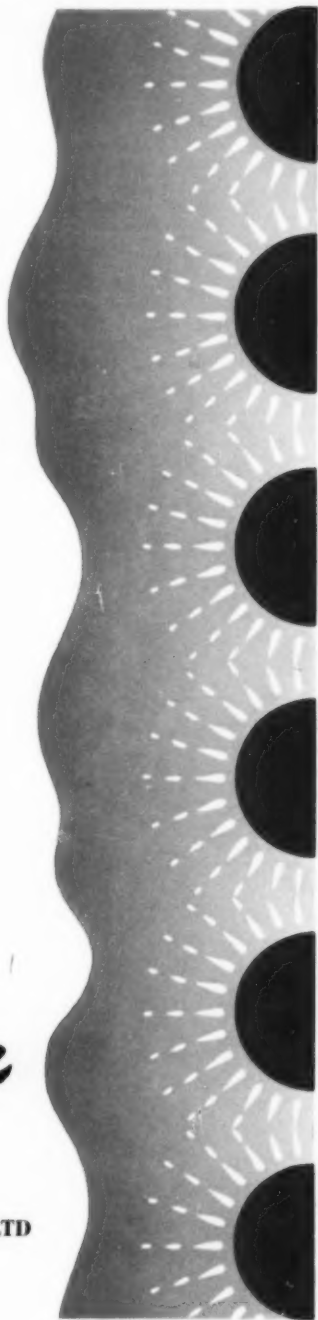


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Abstracts from British and Foreign Journals and Patents

(The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952); while other abbreviations and symbols, together with a list of the periodicals abstracted, will be found at the end of the annual index.)

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Reducing Particle Size. Messinger Bearings.

BP 666,200

A mill which quickly and economically reduces particle size is described. C. O. C.

Apparatus for Emulsifying Liquids. R. J. Jay.

BP 665,981

Drying Fibrous or Granular Material. C. F. Nielsen. BP 665,185

A drying drum contains a screw conveyor, through which fibrous material is blown by a current of heated air. Both drum and screw can be rotated, either to assist the passage of the blown material or to retard it if it requires a more lengthy treatment. J. W. B.

Thread-advancing Reel. Courtaulds. BP 665,732

Supporting Yarn during Withdrawal from Package. a. Abbott Machine Co. BP 665,489

An air pressure difference is established across a yarn package and a previous supporting wall, so that the yarn is pressed against the support and does not collapse during winding from the inside surface of the package. J. W. B.

Carrying Cloth or the like through Drying or similar Chambers. Andrews & Goodrich. USP 2,557,416

When passing cloth or the like through chambers by means of upper and lower guide rollers, the individual rollers are driven so that the speed of each roller is automatically adjusted to the speed at which the cloth is delivered to it. This maintains uniform tension throughout the cloth irrespective of any change in length which the cloth undergoes while passing through the chambers. U.S.P. 2,557,417

The speed at which the cloth is fed to the chamber is automatically adjusted to keep the tension on the cloth constant. C. O. C.

Cylinder Drying Machine. British Cotton Industry Research Assoc. BP 663,161

Over the drying cylinder there is a cover parallel to the surface of the cylinder. At the ends of the cylinder there are seals which restrict admission of air between the cylinder and the cover. Between the front and rear edges of the cover there is a means of sucking air from beneath the cover. The capacity of this suction and the distance of the cover from the material being dried together are such that air is induced to enter at a high velocity with unobstructed passage under the front and rear edges of the cover and to pass over the greater part of the material in contact with the drying cylinder. This ensures continuous rapid removal of the moist air from the surface of the material being dried. C. O. C.

Drying Cloth and Paper. P. Deek. BP 665,238

Hot air is fed under pressure from pivotally mounted and interconnected hollow nozzles on to the cloth or paper. The nozzles taper in plan view from their pivoted parts to their free ends. Elongated slots extend along the nozzles from their pivoted ends parallel to the cloth or paper, so as to direct the hot air on to the material being dried. C. O. C.

Conveying Cloth, Paper, Linoleum, etc. through Full-width Drying or other Treating Machines. J. Dugler. BP 665,305

Braking Device for Web-festooning Apparatus. Patent & Licensing Corp. USP 2,561,165

Indicating the Moisture of Travelling Webs. DuP. USP 2,558,392

An electrical device for controlling the moisture content of warps being can-dried. C. O. C.

Constant-tension Jig. Celanese Corp. of America. USP 2,557,185

A sensing element whose position is determined by the tension on the cloth is connected to a fluid-operated brake so that, as the position of the sensing element changes, the braking force applied to the roller from which the cloth is

being drawn varies inversely with the tension on the cloth. This maintains constant tension on the cloth. C. O. C.

Controlling the Thickness of a Coating applied to a Travelling Web. Haloid Co. USP 2,558,773

A doctor roll having a hard polished surface is mounted opposite the coating roll so as to touch the coating on the web. It can be driven at varying speed so as to regulate the thickness of the coating. A scraper of flexible resilient material extends across the doctor roll so as to clean it before it reaches the coated web. C. O. C.

Single Coating Machine for Flexible Webs. Time. BP 665,891

Furnishing is done by rollers instead of the usual knife spreading arrangement. G. V. S.

Nylon Hose Finishing Machine. British Schuster Co. BP 664,217

A continuous finishing machine has an endless conveyor of leucery forms passing through spray boxes for aqueous impregnation of the hose followed by rubber squeeze rollers and a drying or conditioning chamber in which hot air is circulated. The machine is of the double-ended type with two loading and stripping points in the output. G. E. K.

Hosiery Finishing Machine. Grimsley & Co. (Leicester). BP 664,123

Hose are preboarded or finished by placing them on forms, each pivotally mounted and capable of being manually projected into and out of a closable chamber. Two treatment chambers are arranged side by side with a common sliding door enabling one group of forms to be stripped and reloaded while treatment proceeds in the other chamber. Hot air is drawn through the chamber during the treatment. G. E. K.

Form for Examining and Finishing Stockings. M. Kohlsdorf. USP 2,561,210

Garment Driers. F. W. Howland. BP 664,637

Garment-treating Apparatus. J. C. Shaw and H. E. Blettner. BP 665,353

A flexible porous bag blown up with steam and/or air supports the garment. Means extending longitudinally inside the bag restrict expansion of certain parts while permitting limited ballooning between such parts. C. O. C.

Multiroll Ironing Machines. Lister Bros. BP 664,482

Machines having spreading boards or rollers to stretch the centre of the sheets or the like before they enter into the machine are described. C. O. C.

Producing Patterns Photographically on Copper, etc. Rollers. L. Frank. BP 665,940

Reconditioning Felt Printing Blankets. V. Behrens. BP 664,934

The relaxed felts are steamed and air-dried in the same chamber. C. O. C.

Flame Pistol for Spraying Fusible Powders. Schori Metallizing Process. BP 664,930

A pistol which operates for long periods with powders of m.p. < 1100°C. without requiring cleansing is described. C. O. C.

Compounding, Mixing, and Sheetting of Plastic Materials. James Ferguson & Sons. BP 666,052

Drying Long Fibres (VI p. 133).

Electrostatic Printing (IX p. 135).

II—WATER AND EFFLUENTS

Textile Effluents—Review and Bibliography.

Wesleyan University. Report to New England Interstate Water Pollution Control Commission (1950), 36 pp.; Water Pollution Abn., 24, 187 (Aug. 1951).

A critical review of the literature published on treatment of textile effluents during 1936–1950; 236 references. C. O. C.

PATENTS

Lime and or Soda Treatment of Water. IC1

BP 665,481

Addition of a few p.p.m. of sodium hexametaphosphate to the water increases the proportion of CaCO_3 which crystallises on to existing particles and decreases the number of fresh nuclei obtained. C. O. C.

Mechanical Coagulation of Solids in Liquids. Infillec.

BP 665,433

Removing Sludge from Boilers while Simultaneously Preventing Scale Formation. J. Gulowski.

BP 664,987

In the upper layer of the boiler water there is a floating device for collecting impurities, and in the lower layer perforated conduits. The sludge discharge conduits in both the upper and lower boiler levels contain throttling devices each comprising a chamber containing several perforated diaphragms, the perforations being in staggered relationship and thus acting to change the direction of flow and control the outflow of water and sludge mixture from the boiler. C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS**Bibliography of Lactic Acid and Derivatives.** ORR 413,51* (PB 103,447).

A bibliography dated Feb. 1951 relating to chemical and industrial aspects. C. O. C.

*Photocopies available from T.I.D.U., D.S.I.R., Conard Building, 15 Regent Street, London S.W.1, or (under P.B. No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.—see J.S.D.C., 66, 53 (Jan. 1950).

Chemiluminescence of Vegetable Oils. Y. Tsunoda. *Science (Japan)*, 13, 129 (1943); *Chem. Abs.*, 45, 10,061 (25 Nov. 1951).

The Mg complex salt of phthalocyanine dissolved in various vegetable oils (2 mg./5 c.c.) on heating in a dark chamber yielded green solutions having ruby luminescence. Intensity was greatest in soya-bean oil layered upon glycerol; soya-bean oil and cottonseed oil followed in intensity of luminescence. Purified soya-bean and linseed oils did not luminesce at $< 300^\circ\text{C}$. The luminescence is attributed to peroxide formed in the oil during storage with the complex salt. C. O. C.

Monolayer Study of a Water-soluble Wetting Agent (Nekal BX). K. Schafer. *Kolloid-Z.*, 124, 15-22 (Oct. 1951).

Sodium dibutylnaphthalenesulphonate (Nekal BX), although water-soluble, is able to form stable monolayers if spread on concentrated NaCl solutions (10-125 g./litre). The force-area ($F-A$) graphs obtained from measurements using a Langmuir trough method are found to be reversible for $F < 5$ dynes/cm., but at higher values of F the compression curve differs from the expansion curve and there is a noticeable time-effect, which is negligible, however, after 100 hr. The data obey the equation $FA = aF + b$ (a and b are constant provided F is small), and this equation holds over a wider range as the salt concentration is increased. The magnitude of a indicates that the area occupied by the molecule in the interface is 45-6 sq. Å. in good agreement with the value of 44 sq. Å. obtained by applying the Gibbs adsorption equation to equilibrium surface tension measurements. This agreement shows that the law relating spreading pressure F (surface tension) to concentration c may be written in the form—

$$RT \ln \frac{c}{c^0} = aF + b \ln F$$

where c^0 is a constant, characteristic of the surface-active agent (6-5 mg./litre). This equation differs from the usual Szyszkowski equation. At concentrations > 100 mg. per litre there is a kink in the graphs of F against $\ln c$, which is attributed to the onset of micelle formation. L. P.

Absorption of Dyes on Chromium Hydroxide. S. N. Tewari and S. Ghosh. *Kolloid-Z.*, 124, 31-36 (Oct. 1951).

Three forms of chromium hydroxide are prepared from chromium chloride by precipitation with (a) 10% excess

NaOH, (b) the equivalent amount of NaOH, and (c) 10% less than (b). The washed precipitate is aged for various times (24 hr. to 24 days), and then made up as a suspension of 0.0828 g. Cr_2O_3 per litre. The adsorption by these samples of the basic dyes Methylene Blue and Malachite Green and the acid dye Orange II from solutions of varied concentration is studied at 30° and 60°C . The data given show that the amounts of basic dyes adsorbed are in the order (a) $>$ (c) $>$ (b), whereas those of the acid dyes are (b) $>$ (c) $>$ (a), a fact which is interpreted as evidence of the amphoteric nature of chromium hydroxide. The amounts adsorbed decrease, in all cases, with rise of temperature or increase in age of the precipitate. L. P.

PATENTS

Antistatic Composition. C. C. Wakefield & Co.

BP 665,914

An aqueous solution or dispersion of an ester of a carboxylic acid of mol. wt. < 101 , e.g. diacetin or β -methoxyethyl acetate, and a polyglycol of mol. wt. > 190 , e.g. polyethylene glycol of mol. wt. 4000, is used. C. O. C.

Lubricating and Antistatic Compositions. Courtaulds.

BP 665,475

A clear solution of (a) an alkylamine sulphated oleyl alcohol or alkylamine sulphated oleic acid and (b) a partial ester of a polyhydric alcohol and a fatty acid containing > 9 C in a lubricating oil is a good antistatic agent for cellulose-derivative fibres, particularly acetate rayon. C. O. C.

Sizing Nylon. Monsanto.

BP 666,274

A tough smooth elastic size which does not cut off during weaving consists of an aqueous solution containing (a) an alkali-metal salt of a copolymer of styrene and maleic acid or anhydride, fumaric acid, or an alkyl (1-3 C) ester of either acid or a mixture thereof with styrene or a nuclear-substituted methyl- or chloro-styrene, and (b) a polyethylene or polypropylene glycol. C. O. C.

Fluorinated Aliphatic Phosphates as Emulsifying Agents for Aqueous Polymerisations. DuP.

USP 2,559,749

Compounds of formula $\text{B}-(\text{CF}_2)_n-\text{CH}_2\text{A}$ ($\text{B} = \text{H}$ or F ; $n = 2-4$; $\text{A} =$ a phosphate group) are excellent agents for preparing aqueous dispersions of unsaturated organic compounds preparatory to their being polymerised. C. O. C.

Fluoroalkylsulphuric Compounds—Fluoroalkylating, Detergent, and Flameproofing Agents. DuP.

USP 2,559,751

Compounds of formula $\text{B}-(\text{CF}_2)_n-\text{CH}_2\text{O-SO}_3\text{M}$ ($\text{B} = \text{H}$ or F ; $n = 5-12$; $\text{M} = \text{H}$, alkali metal, or NH_4) are used. C. O. C.

Fluoroalkane phosphonic Compounds. DuP.

USP 2,559,754

Compounds of formula $\text{H}-(\text{CF}_2)_n-\text{CH}_2\text{PO}(\text{OH})_2$ ($n > 0$) are useful dispersing agents and are remarkably stable in acid solution. C. O. C.

Detergents from Acylated Hydroxyalkylamines. Swift & Co.

BP 665,264

Excess of a primary or secondary hydroxyalkylamine, e.g. dipropanolamine or diethanolamine, is treated with an acylating agent of 8-14 C, e.g. coconut oil fatty acids. After the initial condensation a higher fatty acid of > 14 C, e.g. oleic acid, is added to form a hydroxyalkylamine soap with the excess hydroxyalkylamine, and the mixture aged. The product is an excellent detergent with good foaming properties and resistance to hard water. J. W. B.

Water-softening Agent. Monsanto.

BP 665,198

A mixture of finely divided sodium hexametaphosphate (80-96% by weight), sodium carbonate, bicarbonate or percarbonate (19-98-2-0), and an aliphatic monohydric alcohol of 6-12 C (0-02-2-0) has a good rate of solution, and when added to water does not tend to mat or sink to the bottom of the vessel. C. O. C.

Water-softening Agent. Monsanto.

BP 665,455-7

The alcohol is replaced by an amine of formula $\text{R}^1\text{-NH-R}^2$ ($\text{R}^1 = \text{H}$ or an aliphatic or alicyclic hydrocarbon radical; $\text{R}^2 =$ an aliphatic or alicyclic hydrocarbon radical; at least one of these two radicals contains 6-10 C), e.g. heptylamine, by "propylene polymer" (mainly dodecene) or octene, or by an unsubst. aliphatic carboxylic acid containing 6-10 C atoms for each polar group. C. O. C.

Lubricating, Conditioning, and Antistatic Agents.

BrC. *BP* 664,415
A mixture of a mineral oil, castor oil, a nuclear-alkylated phenol, a long-chain aliphatic acid, a hydroxyalkylamine, and the product obtained by treating a long-chain aliphatic acid, a vegetable oil, and a mineral oil with H_2SO_4 , neutralizing with alkali and a hydroxyalkylamine, desalting, and dehydrating, is especially suitable for use on acetate rayon. *C. O. C.*

Polymeric Tris- β -hydroxyethyl Cyanurate for Finishing Textiles.

USP 2,557,625
The polymer obtained by heating tris- β -hydroxyethyl cyanurate at 105–150°C. is useful for modifying the texture and handle of textiles. *C. O. C.*

Solvent for Acrylonitrile Polymers.

DuP. *BP* 665,004
A mixture of nitromethane (55–96.5% by weight) and water (45–3.5) is a solvent and swelling agent for polymers containing not < 85% by weight of acrylonitrile. *C. O. C.*

Trimethylisothiocyanatosilane — Water-repellent Agent.

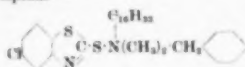
General Electric Co. *USP* 2,559,340
Compounds of formula $(CH_3)_3Si(NCZ)_n$ ($n = 1-3$; $Z = O$ or S), e.g. trimethylisothiocyanatosilane, are useful for rendering materials water-repellent without formation of hydrogen halide by-products. *C. O. C.*

Water-repellent Composition stabilised with a Tertiary Heterocyclic Aromatic Amine.

DuP. *USP* 2,557,653
A water-repellent composition consisting of an alcohol dispersion of quaternary pyridinium salts of a hydroxy-methylmelamine-fatty hydroxymethylamide condensation mixture (obtained by condensing a hydroxymethylamide of a fatty acid with 4.5–36.0% by weight of hydroxy-methylmelamine in an alcohol solution of 0.5–4.0 mol. of HCl per mol. of hydroxymethylmelamine) is stable when diluted with water if the composition contains 2.5–12% of free tertiary heterocyclic aromatic amine of 5–10°C. e.g. pyridine, and 40–50% of the condensation mixture. *C. O. C.*

Benzyldimethylammonium 5-Chlorobenzo-2-thiazolyl Sulphide — Mildewproofing Agent.

R. T. Vanderbilt Co. *USP* 2,557,172
Benzyldimethylammonium 5-chloro-2-benzo-thiazolyl sulphide—



is a powerful fungicide useful for mildewproofing textiles by applying 0.01–0.5% of it from 50–25% aqueous acetone. It resists leaching, washing, and dry cleaning. *C. O. C.*

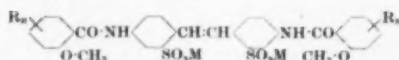
Aluminium Carboxylic Acid Soap Heavy-metal Salt of Hydroxyquinoline—Fungicidal Composition.

Scientific Oil Compounding Co. *USP* 2,561,379
A "heavy-metal" soap, especially a Zn or Ni soap, or an Al soap is heated with Cu 8-quinolineoxide until a homogeneous composition is formed. This composition is highly dispersed or dissolved in petroleum solvents, and applied to textiles it imparts a fungicidal finish which is highly resistant to leaching and washing. *C. O. C.*

USP 2,561,380
A water-insoluble soap, especially a Zn, Cu, or Ni soap, is heated with copper 8-quinolineoxide or an alkaline earth, heavy metal, or Al salt of a hydroxyquinoline. *C. O. C.*

Blue-fluorescent Agents for Optical Bleaching.

DuP. *BP* 665,415
Compounds of formula—



($R =$ a methoxy radical attached in the 4- or 5-position; $m = 1$ or 2 ; $M =$ a cation) are substantive to cellulose and give a bluish fluorescence under ultraviolet radiation. *C. O. C.*

Optical Brightening Agents. GY.

Compounds of formula— *BP* 666,198
$$Y-NH-\text{CH}-CH-\text{CH}-CH-A$$

$$SO_3H \quad SO_3H$$

($Y =$ "carbacyl" or 1:3:5-triazine radical; neither radical may contain a chromophore; and the ring A and, if desired, also Y may contain substituents which cannot displace the region of absorption into the visual range) have good affinity for cellulose, protein, and nylon fibres and give an intense greenish-blue to green fluorescence, which makes them particularly suitable for use on wool. *C. O. C.*

Wax Composition for Coating or Impregnating Fibrous Materials.

Monsanto. *BP* 664,778
Polystyrene which has been alkylated (0.2–66) by treatment with polymeric propylene when mixed with a hydrocarbon wax (99.8–40) yields a homogeneous blend of improved tensile strength, elongation, hardness, gloss, and adhesion to paper. *C. O. C.*

Aqueous Acid Fluoride Rust-removing Compositions.

Pennsylvania Salt Manufacturing Co. *USP* 2,558,013
Addition of 1–5% of a water-soluble aliphatic compound containing a hydrophilic oxygen-containing group (other than $COOH$) as its principal functional group, e.g. diethylene glycol monobutyl ether, greatly improves the wetting power of aqueous acid fluoride solutions. *C. O. C.*

Aqueous Colloidal Dispersions of Polymers (VI p. 133).
Silicon-containing Rubber Compositions (XIII p. 139).
Polymeric Triazine Derivatives (XIII p. 139).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS**Absorption Bands of Benzene and its Derivatives in the Visible Region.**

K. Shibata, H. Kushiya, and S. Mori. Bull. Chem. Soc. Japan, 24, 188–189 (Sept. 1951).

The absorption spectra of benzene, chlorobenzene, toluene, and tetralin show a weak band in the region 5200–5300 Å, which has not been previously reported. It is considered that this may be due to singlet-triplet transitions which are not considered in the Goepfert-Mayer-Sklar theory (*J. Chem. Phys.*, **6**, 645 (1938)), although the agreement with calculated energy differences is not satisfactory. *A. J.*

Co-ordination of Organic Bases with Palladium (II) Cyanide.

F. Feigl and G. B. Heisig. J. Amer. Chem. Soc., 73, 5631–5633 (Dec. 1951).

Palladium (II) cyanide co-ordinates ammonia and many organic nitrogen bases, e.g. pyridine, ethylenediamine, quinoline, to form crystalline compounds which are homologues of the diamine, β -naphthylamine forming a homologue of the monoamine. The reaction is one between a Lewis acid and base respectively. Many amines co-ordinate with palladium (II) cyanide to form adsorption complexes (lakes). In the case of acid-base indicators such lakes have a similar colour to the acid solution of the indicator. An explanation is proposed based on resonance and the Lewis theory of acids and bases. The coloured oxidation products of colourless amines form lakes with palladium (II) cyanide, and two explanations for their formation are suggested. *C. O. C.*

Electric Moments of Some Azo Dyes.

T. W. Campbell, D. A. Young, and M. T. Rogers. J. Amer. Chem. Soc., 73, 5789–5791 (Dec. 1951).

The electric dipole moments of a series of azo dyes—



($X = H, CH_3, C(CH_3)_3, O-CH_3, Cl, I, SCN, SeCN, or NO_2$) were measured in benzene at 25°C. and compared with values calculated from bond measurements. It was found that when X is a strong electron acceptor ($NO_2, SeCN, or SCN$) or Cl or I , the observed moment was larger than the expected value. These results are interpreted in terms of resonance theory. *C. O. C.*

Synthesis of Naphthalene Dyes used in Chemotherapy. A. M. Armstrong. *Actas y Trabajos Congr. peruano quim.*, 7. Congr. (Lima, Peru), 1, 243-251 (1949); *Chem. Abs.*, 46, 102 (10 Jan. 1952).

Protosol soluble is prepared by sulphonating β -naphthylamine in two steps, treating with NaOH, acetylation, and coupling with diazotized sulphamylamide. Aseptol Red (8-acetamido-2-*p*-sulphamylphenylazo-1-naphthol-3,6-disulphonic acid) is prepared in a similar fashion. C. O. C.

Hydroxyquinones. VIII—Structure of Droserone 1. IX—Structure of Droserone 2. X—Pigment of Drosera peltata. M. Asano and J. Hase, *J. Pharm. Soc. Japan*, 63, 93-96, 410-411 (1943); *Chem. Abs.*, 46, 92 (10 Jan. 1952).

VIII and IX—Droserone, a dye obtained from *Drosera Rotundifolia*, is shown to be 3,5-dihydroxy-2-methyl-1,4-naphthoquinone.

X—The roots of Drosera peltata contain plumbagin (5-hydroxy-2-methyl-1,4-naphthoquinone) as well as droserone. C. O. C.

Coloured Condensates of 4-Chloroquinoline and of Phthalonitrile with Heterocyclic Molecules containing an Active Methylene Group. A. Meyer and G. Bouchet. *Compt. rend.*, 229, 372-374 (1949); *Chem. Abs.*, 46, 113 (10 Jan. 1952).

Coloured compounds intermediate between indigoids and cyanines are obtained by condensing 4-chloroquinoline in pyridine with indoxyl (I), oxindole (II), 3-hydroxythianaphthen (III), and 3-coumaranone (IV) to yield the following 1:4-dihydroquinolines: 4-(3-keto-2-indolylidene) brick red; 4-(2-keto-3-indolylidene) brownish red, blue in alkaline solution, orange in acid solution; 4-(2,3-dihydro-3-keto-2-thianaphthylidene) dark red; 4-(2,3-dihydro-3-keto-2-benzofuranylidene) yellowish brown. These products dye acetate rayon from aqueous dispersion. Indigoid dyes were prepared by condensing 1,3(2H, 4H)-isquinolinedione in acetic anhydride with heterocyclic compounds containing a reactive CH_2 group. I, III, and IV yield the following 1:3(2H, 4H)-isquinolines: 4-(3-keto-2-indolylidene) dark violet; 4-(2,3-dihydro-3-keto-thianaphthylidene) yellowish red; 4-(2,3-dihydro-3-keto-benzofuranylidene) yellow brown; but these compounds do not change in hue with variation in pH nor do they dye acetate rayon. C. O. C.

Kinetics of Formation of Adsorption Films of Thymol Blue. H. C. Saraswat and A. Singh. *Kolloid-Z.*, 123, 100-104 (Aug.-Sept. 1951).

Using a Langmuir trough, with which an accuracy of 0.02 dyne/cm. could be attained, in conjunction with a dynamic maximum bubble pressure method of measuring the surface tension of alkaline solutions of Thymol Blue (0.00006-0.001 M.), the authors have studied the rate of formation of the surface film. They show that in the equilibrium state the film has a gaseous expanded form of 5.6×10^{15} mol./sq. cm. (179 sq. Å./mol.) at zero pressure. The gaseous character of the film and its slow formation are attributed to a potential barrier caused by the ionised end-groups in the Thymol Blue molecule. L. P.

Behaviour of some Homologues of Agfacolor Image Dyes in Aqueous Solutions. K. Meyer and A. Bettesch. *Z. phys. Chem.*, 45, 226-233 (1950); *Chem. Abs.*, 46, 45 (10 Jan. 1952).

A series of 1-*p*-sulphophenyl-3-alkyl-4-*p*-diethylamino-phenylimino-5-pyrazolones, with the alkyl group varying from 1 to 15 C atoms, was prepared by condensing the corresponding pyrazolones with *p*-nitro-N,N-diethyl-aniline in absolute ethyl alcohol in presence of triethylamine or piperidine. They were all readily soluble in alcohol and showed practically identical absorption curves. C. O. C.

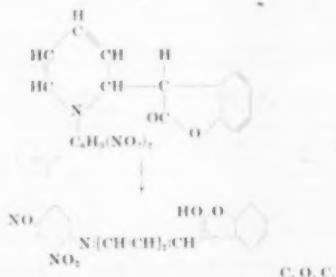
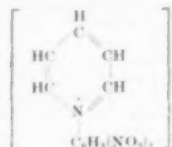
Metachromasy of Thiazine Dyes produced by Chondroitin Sulphate. A. Levine and M. Schubert. *J. Amer. Chem. Soc.*, 74, 91-97 (Jan. 1952).

Spectrophotometric study of the appearance and disappearance of metachromasy in solutions of two thiazine dyes as a function of increasing chromotropic concentrations rules out the possibility that metachromasy is due to dye dimerisation, but the results can be interpreted to offer slender support to the theory of dye polymerisation. It is suggested that the effect may be due to loose binding

of dye cations with chromotropic polyanions, as adsorption of dye ions on colloidal surfaces may change their colour. C. O. C.

New Polymethine Dyes. P. Pfeiffer and E. Enders. *Chem. Ber.*, 84, 313-318 (1951); *Chem. Abs.*, 45, 9047 (25 Oct. 1951).

When *p*-nitrobenzoyl chloride is treated with isocoumarone in pyridine, a deeply coloured compound results. Study of this reaction and of the formation of polymethine dyes by similar procedures suggests that cleavage of the pyridine ring with an activated CH_2 group may be explained as follows—

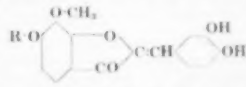


New Synthesis of Flavones. N. Narasimhaiahari and T. R. Seshadri. *Proc. Indian Acad. Sci.*, 30 A, 151-162 (1949); *Chem. Abs.*, 45, 10,242 (25 Nov. 1951).

Iodine in alcoholic Na acetate is a convenient reagent for converting hydroxyflavanones to flavones. If the OH is absent or if the reaction is conducted hot, mixtures of flavones and chalcones are formed. The method is also applicable to glycosides. C. O. C.

Anthochlor Pigments. VIII—Pigments of Cosopsis grandiflora Nutt. III. T. A. Geissman and W. Moje. *J. Amer. Chem. Soc.*, 73, 5765-5768 (Dec. 1951).

That leptosidin and its glucoside leptosidin have the structure—



(R = H (leptosidin) or $\text{C}_6\text{H}_4\text{O}_2$ (leptosin)) has been confirmed by their total synthesis. C. O. C.

Light-exposure Tests on Chrome Yellow. R. Haug. *Deutsche Farben-Z.*, 5, 343-348 (1951); *Chem. Abs.*, 46, 276 (10 Jan. 1952).

A chrome yellow (I) of poor fastness to light and containing 82% PbCrO_4 and 15.5% PbSO_4 and a 4 : 1 mixture of I and zinc white were coated on paper and exposed to sunlight and selected wavelengths by means of filters. Exposure was under glass, but air was given free access to the coatings. After approx. 2800 kilolux-hr. there was little change in hue, but brightness fell off where exposure had been to light of 300-700 m μ . Zinc white had no protective effect. In evacuated tubes (attached to P_2O_5 tubes) the effect was the same, but the mixture lost less brightness than the chrome yellow alone. Seven other chrome yellows showed effects of varying intensity, rhombic chrome yellow being less fast to light than the monoclinic variety. C. O. C.

Origins of Pigment and Varnish Techniques. E. Crivelli, *Ind. Vernice* (Milan), 5, 134-138 (1951); *Chem. Abstr.*, **46**, 275 (10 Jan. 1952).
A survey of the development of pigments, coatings, etc. from ancient times. C. O. C.

Lead Cyanamide as a Rust-inhibiting Pigment. E. Stock and H. Blum, *Deutscher Farben-Z.*, **5**, 317-321 (1951); *Chem. Abstr.*, **46**, 275 (10 Jan. 1952).
An account of its manufacture and properties, and of tests made with it in different vehicles. C. O. C.

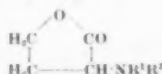
PATENTS

Use of Irradiation with High-speed Electrons to Effect Chemical Reactions. Electronised Chemicals Corpn. BP 665,263

Many chemical operations normally requiring heat and/or pressure and/or catalysts, e.g. oxidation, halogenation, nitration, hydrogenation, vulcanisation, etc., may be carried out under normal pressure and without heat or a catalyst by irradiating the reaction mixture with electrons having a velocity equivalent to > 1000 kv. In some cases it is preferable to use moderate heat and/or pressure and certain contact materials, e.g. MgO, ZnO, or Al_2O_3 . The process can be used for making dyes. Thus pure indigo is obtained by high-speed electron bombardment of indican or indoxyl-sulphuric acid; all kinds of diazo compounds can be transformed into azo dyes; irradiation of suitable phenols either alone or in conjunction with amines or diazotised amino compounds yields colouring matters by facilitating coupling; irradiation of transparent phenols, e.g. those used in Bakelite manufacture, transforms them into a red dye. C. O. C.

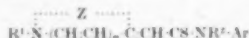
Diazotized Material. General Aniline. USP 2,560,137
2,7-Dihydroxynaphthalene-3,6-disulphonic acid and the corresponding sulphonamides used as coupling components with light-sensitive diazo compounds of the *p*-phenylenediamine series yield prints of high colour value, of good fastness to light and washing, and stable to precoupling and discoloration of the background after exposure. C. O. C.

α -Amino- γ -lactones. BASF. BP 659,483
 α -Amino- γ -lactones—



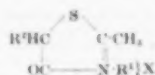
(R^1 and R^2 = the same or different Alk., Ar., etc.) are prepared by condensing α -halogenobutyrolactones with a secondary amine in presence of an acid-binding agent. Thus α -chlorobutyrolactone is heated for 8 hr. at 130°C . with 50% aq. dimethylamine. R. K. F.

***N*-Acylated Arylamino-2-thioethylidene Derivatives of Heterocyclic Bases—Intermediates.** Eastman Kodak Co. USP 2,556,516
Compounds of formula—



(R^1 = Alk., or Ar when Z gives a benzothiazoline or a thiazolizidine nucleus; n = 0 or 1; R^2 = an aliphatic acyl group; Z = atoms to complete a heterocyclic nucleus of the benzothiazoline, benzoxazoline, α - or β -naphthothiazoline, dihydroquinoline, 4-thiazoline, thiazolidine, and 3,3-dimethylindoline series) are obtained by condensing an aryl isothiocyanate with a cycloammonium quaternary salt containing an active CH_2 group. Condensed with phenacyl bromide they yield *N*-arylthiazolizidine dyes, and with ethyl bromoacetate they yield methin dyes containing a 3-aryl-4-thiazoline nucleus. Treated with "alkyl salts" they yield β -acylated arylamino- β -alkylmercaptovinylcycloammonium quaternary salts which condense with cycloammonium quaternary salts containing a reactive CH_2 group to yield trinuclear cyanine dyes. C. O. C.

3-Substituted 4-Keto-2-methylthiazolinium Dye Intermediates. General Aniline. USP 2,561,245
Compounds useful as cyanine dye intermediates for making trinuclear cyanine dyes have the formula—



(R^1 = Alk., cycloalkyl, Ar, aralkyl, or heterocyclic; R^2 = H or CH_3 ; X = anion). C. O. C.

Aromatic and Heterocyclic Diazosulphonates. General Aniline. BP 663,745

Diazosulphonates $R^1\text{-N}(\text{N}=\text{SO}_2)\text{-R}^2$ (R^1 = aryl substit. with arylamino, benzylamino, dialkylamino, or heterocyclic-amino; R^2 = aminosulfonyl, alkoxyaryl, acylaminosulfonyl, or a heterocyclic radical) are prepared (a) by treating a diazonium compound with a sulphonyl chloride $R^2\text{-SO}_2\text{Cl}$, or (b) by condensing a diazonium salt, a diazocyanide, or a diazoamino compound with a sulphonic acid $R^2\text{-SO}_3\text{H}$. Unlike the related diazosulphonic acids $R^1\text{-N}(\text{N}=\text{SO}_3\text{H})$, they will couple with azo dye coupling components in absence of ultraviolet radiation. Many of those which contain strongly electropositive substituents are decomposed by ultraviolet radiation, and so may be used as the light-sensitive element in diazotype printing. Thus, a diazo solution prepared from *p*-diethylaminoaniline is added to an alkaline solution of aniline-*p*-sulphonic acid in the cold. The diazosulphonate—



separates as an orange precipitate. E. 8.

Diazotisation of Esters of Aminobenzoic Acids. Bozel-Maletra, Société Industrielle des Produits Chimiques. BP 663,870

Esters of aminobenzoic acids can be diazotized in absence of alcohol and without saponification by starting the reaction at $> -18^\circ\text{C}$. Thus, all parts being by weight, methyl anthranilate sulphate (50) is added to a mixture of ice (100), H_2SO_4 (22), and water (10), so that the temperature falls to about -20°C . A solution of sodium nitrite (15) in water (30) is quickly added whilst stirring. Sodium sulphate separates as the temperature rises, and the methyl anthranilate sulphate passes into solution as diazotisation proceeds. E. 8.

Colour Couplers from Bifunctional Intermediates having Aliphatic and Aromatic Amino Groups. Kodak. BP 664,580

Colour couplers are obtained by acylating the aliphatic amino group of an aminophenylalkylamine with an ester and then acylating the aromatic amino group with another acylating agent, either the ester or the second acylating agent having a coupling function, i.e. a group which reacts with the oxidation product of a primary aromatic amino developing agent. C. O. C.

1-Amino-4-bromoanthraquinone-2-sulphonic Acid. BrC. BP 659,684

Bromine (or chlorine) is introduced in a stream of air or inert gas into a solution containing 3% by weight of 1-aminoanthraquinone-2-sulphonic acid and 3% NaCl at 0°C . until the amount required for monobromination has been added. The bromine-air mixture is made by passing dry air at 150°C . through a vessel containing bromine at 10 – 25°C . R. K. F.

Solubilised Azoic Dyes. Ciba. BP 662,573

Insoluble azoic dyes of the type aromatic amine- α -aryl- amide of 2-hydroxy-3-naphthoic acid (or of other suitable hydroxycarboxylic acids) can be solubilised by treatment with the carboxylic acid chloride of chloro (or bromo)-sulphoacetic acid $\text{Cl-CO-CH}_2\text{SO}_3\text{H}$ (X = Cl or Br). Acylation of the azoic dye occurs on both the OH and the CO-NH of the coupling component, giving products which are in general readily soluble in water. They are relatively stable to weak acids, but with weak alkalis the insoluble dye is regenerated. Hence they may be dyed or printed on wool as well as on cotton, and the colour developed on the fibre by simple treatment with cold dilute ammonia or with ammonia vapour. Thus, all parts being by weight, chlorosulphoacetic acid (60), prepared from 1 mol. of chloroacetic acid and 1-1 mol. of SO_3 , is mixed with dry pyridine (400), and phosgene (36) passed in at 30 – 40°C . The azoic dye (35) 2,5-dichloroaniline-2-hydroxy-3-naphtho- α -amide is added, and the mixture stirred at 70 – 75°C . until completely soluble in water. The product

is isolated by acidifying with dil. H_2SO_4 and salting out. It may be applied to wool from a boiling acid bath, and the scarlet colour regenerated on the fibre by adding sodium acetate and boiling. Or cotton may be printed with a suitably thickened paste of the solubilised dye containing acetic acid, and after steaming, the insoluble colour developed in 1–2% aq. ammonia. E. 8.

Blue, Chromiferous, Monoazo Dyes for Wool, etc. Ciba. BP 665,372

Blue dyes for wool, nylon, etc. are made by chroming in substance monoazo compounds made by diazotising a monosulphonic acid of a 2:5-dialkoxyaniline and coupling with an *ortho*-coupling naphthol or naphtholsulphonic acid. Thus, by treating the monoazo compound 2:5-dimethoxyaniline-4-sulphonic acid-1 naphthol-5-sulphonic acid with a solution of basic chromium sulphate $CrOHSO_4$ at 120–125°C. in a lead-lined autoclave, a blue acid dye is produced. E. 8.

Monoazo Metachrome Dyes. 8. BP 665,627

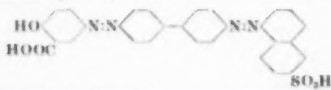
Mainly red metachrome dyes are made by diazotising a sulphonylamide of α -aminophenol (aryl = phenyl, chlorophenyl, tolyl, anisyl, or phenetyl) and coupling with the amide, methylamide, or ethylamide of 1-phenyl-5-pyrazolone-3-carboxylic acid. Thus 2-aminophenol-4-sulphonylamide is diazotised and coupled with 1-phenyl-5-pyrazolone-3-carboxylic acid in presence of ammonia. The product dyes wool yellow red by the afterchrome or neutral metachrome process. E. 8.

Azo Dyes for Acetate Rayon, Nylon, etc. YDC. BP 664,258

Monoazo dyes of the type $R^1-NR^2-SO_2-Arylene-NH_2(p)$ (N -hydroxyalkylated aromatic amine (R^1 and R^2 = H, alkyl, hydroxyalkyl, or together = alkylenes) give bright hues on nylon as well as on cellulose acetate. If the coupling component is a derivative of α -naphthylamine, the dyes on acetate rayon have poor fastness to light, although those on nylon have good fastness. Thus the dye sulphonylamide $\rightarrow N$ -hydroxyethyl α -naphthylamine gives bright scarlets of good light fastness on nylon, but of poor light fastness on cellulose acetate. E. 8.

Disazo and Trisazo Dyes for Leather, etc. General Aniline. BP 665,049

Desamination of amino-disazo and -trisazo compounds derived from tetrazotised benzidine, or its methyl, methoxy, or sulpho derivatives, gives red and brown dyes which penetrate leather more deeply than the corresponding undesaminated compounds. Some of them also give useful results on wool-cotton, etc. mixtures. Desamination is accomplished by diazotisation of the amino-disazo or -trisazo compound followed by reduction with sodium acetate or, preferably, sodium formate. Thus, all parts being by weight, benzidine (184) is tetrazotised, and coupled first with salicylic acid (145) and then with 1-naphthylamine-7-sulphonic acid (230), in presence of soda ash. Hydrochloric acid (620) and sodium nitrite (75) are then added at 15–20°C. to effect diazotisation, followed by sodium formate (125). After 1 hr. at 70°C., the desaminated product—



is separated by filtration.

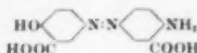
E. 8.

Substantive, Copperisable, Tetrakisazo Dyes. Ciba. BP 662,253

Substantive dyes which may be coppered on the fibre are made by coupling 1 mol. of 3:5'-dihydroxy-2:2'-dinaphthylamine-7:7'-disulphonic acid with 1 mol. each of two different diazotised aminoazobenzene acids, of formula—



respectively (R^1 and R^2 = phenyl residues containing OH and COOH *ortho* to each other). Thus, the monoazo compound 2-nitro-5-aminobenzoic acid-salicylic acid is reduced to the aminoazobenzene-carboxylic acid—



which is diazotised and coupled with 3:5'-dihydroxy-2:2'-dinaphthylamine-7:7'-disulphonic acid in presence of soda ash. Caustic soda is then added, followed by the diazo compound from the aminoazo dye 5-aminosalicylic acid- α -resorcinol. The resulting tetrakisazo dye gives reddish blue on cotton when aftercoppered. E. 8.

Nitrosulphamylidiphenylamines. BFC. BP 659,172

Nitrodiphenylamines containing a sulphamyl group (presumably dyes for cellulose acetate) are prepared by condensing aniline, which may be substituted in the nucleus, with a halogenonitrobenzenesulphonamide. Thus aniline and *p*-chloro-*m*-nitrobenzenesulphonamide are refluxed in water containing sodium acetate and a small quantity of sulphated lauryl alcohol. R. K. F.

Polyaminopolyarylimethane Dyes of the Rosaniline Type. American Cyanamid Co. BP 665,322

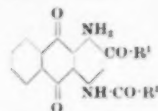
A dispersion of the leuco-polyaminopolyarylimethane is treated in excess of arylamine with gaseous O_3 in presence of a vanadium catalyst. High purity and increased yield are obtained by this method of oxidation, which can be carried out as a continuous process. C. O. C.

Air-free Indigo Paste. DuP. USP 2,550,269

A cheap way of producing high-density, air-free indigo paste is to filter the aq. alkaline indigo slurry, wash the filter cake until free from alkali, pass steam through it until all the displaceable water is removed, and finally add water to the cake to produce a paste of the desired strength. C. O. C.

4-Acylamino-1-amino-2-anthraquinonyl Aryl Ketones—Vat Dyes. Ciba. BP 663,383

Reddish-blue vat dyes, fast to light, chlorine, and water spotting, and of formula—



(R^1 = aromatic residue of 6–12 C; R^2 = aromatic residue of < 13 C) are prepared by acylating an aryl 1:4-diamino-2-anthraquinonyl ketone (BP 661,045). Thus, 1:4-diamino-2-*p*-chlorobenzoylanthraquinone is heated at 110–116°C. in *o*-dichlorobenzene with *p*-chlorobenzoyl chloride and pyridine. R. K. F.

Acylaminoisophthaloylaminoanthraquinones—Vat Dyes. Ciba. BP 659,021

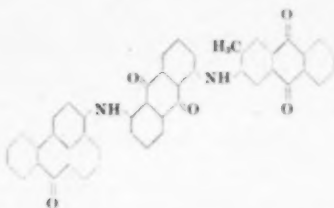
Non-tendering isophthaloylaminoanthraquinone dyes are prepared by condensing 2 mol. of a 1-aminoanthraquinone with 5-nitroisophthaloyl dichloride, reducing, and subsequently acylating the resulting 5-amino group. Thus, 1-aminoanthraquinone is condensed in nitrobenzene with 5-nitroisophthaloyl dichloride. After reducing, by vatting with aq. NaOH and hydrosulphite, the resulting compound is acylated by refluxing with *m*-methylsulphonylbenzoyl chloride in nitrobenzene. R. K. F.

Sulphonated Aryloxybenzanthrimides—Wool Dyes. 8. BP 660,550

Orange to brown neutral-dyeing wool dyes, fast to washing, milling, and light, are prepared by condensing a 3-halogenobenzanthrone with a 1-aminoanthraquinone carrying an aryloxy group in any position but the 2-position, and finally sulphonating. Thus, 1-amino-3-phenoxyanthraquinone and 3-bromobenzanthrone are heated with potassium acetate and cuprous chloride in nitrobenzene at 200°C. The resulting product is sulphonated with 9% oleum. R. K. F.

Benzanthrone-Acridine Vat Dyes. DuP. BP 660,385

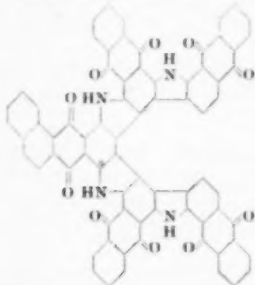
A 3-halogenobenzanthrone is condensed with a 1:5-diaminoanthraquinone, and the resulting product further condensed with an anthraquinone derivative having halogen and methyl *ortho* to one another to give compounds of the form—



On fusion with alkali these compounds form grey vat dyes containing probably two acridine rings. Thus, the product formed by condensing 3-bromobenzanthrone and 1:5-diaminanthraquinone in nitrobenzene is further condensed with 2-bromo-3-methylanthraquinone, all in presence of Na_2CO_3 and copper acetate at 170–210°C. The compound so formed is fused with methyl alcoholic KOH at 145°C. R. K. F.

Benzanthraquinone Carbazole Vat Dyes. Ciba.

5:8-Dihalogeno-1:2-benzanthraquinones are condensed with 4-amino-1:1'-disanthrimides, and the products cyclized in a mixture of aluminum chloride and pyridine, to yield grey vat dyes, containing four carbazole rings, of formula—



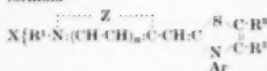
R. K. F.

NN'-Diarylperylene-3:4:9:10-tetracarboxydimides—Vat Dyes. General Aniline.

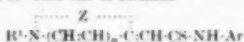
Perylene-3:4:9:10-tetracarboxylic acid or its dianhydride is condensed with an arylamine in water. Thus, the acid is first dissolved in dil. aq. KOH and precipitated in a fine form by pouring into dil. acetic acid containing *p*-anisidine. The whole is then heated at 140–145°C. for 5 hr. R. K. F.

N-Arylthiazole Monomethine Cyanine Dyes. Kodak.

Dyes of formula—

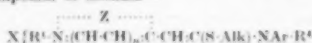


(Z = atoms to complete a benzothiazole, benzoselenazole, α - or β -naphthothiazole, quinoline, thiazolone, thiazole, or 3:3-dimethylindoline nucleus; R^1 = Alk or, when Z yields a benzothiazole or thiazolone nucleus, Ar; R^2 = Alk or Ar; R^3 = H, alk, or Ar; n = 0 or 1; X = an anion) are obtained by condensing a compound of formula $\text{R}^2\text{CHX}\cdot\text{CO}\cdot\text{R}^3$ with one of formula—



BP 665,289

A compound of formula—



(R^4 = an aliphatic acyl group) is condensed with a cycloammonium quaternary salt having a reactive CH_2 group in the 2- or 4-position to the quaternary N-atom. C. O. C.

β -Substituted Polymethine Dyes. Gevaert.

Symmetrical or unsymmetrical β -substituted polymethine dyes are prepared by condensing compounds of formula—



(R^1 = Alk, aralkyl, Ar, carboxyalkyl, or a subst. or unsubst. trimethylene bridge, one end of which is linked to the *peri* C atom of the nucleus completed by Y; R^2 = S-Alk, S-Ar, Se-Alk, O-Alk (Alk subst. or unsubst.), or radicals obtained by substitution for Hal in the R^3 position, e.g. 2-mercaptobenzothiazole; Y = atoms to complete a 5- or 6-membered heterocyclic ring; X = an acid residue) with a cycloammonium salt, an intermediate, or an aldehyde containing a group which reacts with the CH_3 group. C. O. C.

Isolating Quercitrin from Monkey Nut Shins. Administrator of the U.S. Federal Security Agency.

Carbon Black by Thermal Decomposition of Carbonaceous Fluids. P. H. Royter. USP 2,557,143

A process whose chemistry is similar to that of the thermatomic process is described. C. O. C.

Carbon Black. J. W. Woodward. BP 665,583

Blacks of good quality are cheaply obtained in high yield by the direct incomplete combustion of liquid carbonaceous materials, e.g. waste oils. C. O. C.

Chromic Oxide. C. K. Williams & Co. USP 2,560,338

Sodium chromate is treated with sodium thiosulphate in aqueous solution in presence of such amount of water as will yield a solid, dry-to-moist product. The reaction mass is stirred as it thickens and solidifies. The product is formed as particles and is calcined. The hue of the pigment may be varied by adjusting the pH of the mass between 7 and 10 during agitation, the lightest product being obtained if pH 7 is used. C. O. C.

γ -Ferric Oxide Hydrate and γ -Ferric Oxide. Columbian Carbon Co. USP 2,560,970-1

A stoichiometric proportion of an alkali or alkaline-earth metal hydroxide is added quickly and with continuous stirring to a 15–20% aq. soln. of ferrous sulphate at 180–200°C. The resulting thick slurry becomes quite thin if kept at 180–200°C. for 10–15 min. Then it is run into approx. four times its volume of 0.5–2.0% aq. ferrous sulphate at 60°C., so as to dilute the slurry and simultaneously to bring it to 75–85°C. It is kept stirred, and air is blown through it for 2 hr., when a clean orange-yellow pigment is obtained, which is filtered, washed, and oven-dried at 230°C. On heating to 450–500°C. it is dehydrated to the brown γ -ferric oxide. Increased yield and particle size may be obtained by heating the cooled, oxidized slurry to 135–150°C., adding scrap iron every 24 hr., oxidizing for 3–5–4–5 days, and finally drying at 250°C. C. O. C.

Transparent Iron Oxide Pigments. American Cyanamid Co. USP 2,558,302

Crystalline iron oxide containing not more than 5–7% of free water, and < 1% combined anion of average particle size < 0.1 μ , maximum diameter, and of visibility value T_D/T_T not < 75 for unscattered transmitted light when measured in film pigmented with it and having a wet thickness of approx. 0.005 in. (T_D for wavelength of 400–700 m μ . = amount of light transmitted undeviated; T_T = sum of amounts of light transmitted but scattered and light transmitted undeviated), is obtained by adding an aqueous solution of a ferric salt to an aqueous alkali stoichiometrically equivalent to the ferric salt. The solutions are at 40–90°C. and the resulting slurry is digested for not < 15 min. at 40–95°C. The precipitated iron oxide is washed with water until it contains < 1% water-soluble matter, and is finally dried at < 140°C. USP 2,558,303

A product of more uniform particle size is obtained by adding an aqueous solution of a ferrous salt to at least its stoichiometric equivalent of aqueous alkali-metal carbonate at < 40°C., and oxidizing at < 40°C. until all the iron is in the ferric state, the time and temperature of oxidation being controlled so as to yield a pigment of the

viscosity value described in the previous patent. Oxidation is carried out in presence of not $< 0.1\%$ on the weight of the iron calculated as $Fe_2O_3 \cdot H_2O$, of a water-soluble silicate or salt of Zn, tartaric, citric or tannic acid, or a water-soluble salt of such acid.

USP 2,558,304

Light yellow to brownish-orange pigments are obtained if a water-soluble ferrous salt is added to at least 130% of its stoichiometric equivalent of an alkali-forming metal hydroxide dissolved in water at $< 40^\circ C$, the slurry then being oxidized as described above.

C. O. C.

Titanium Dioxide. DuP.

USP 2,559,638

Modification of USP 2,488,439 (U.S.P.C. 66, 337 (1950)). If the oxidation of the $TiCl_3$ is carried out in presence of a small amount of $AlCl_3$, neutralisation can be omitted, and the residual halogen present in the TiO_2 precipitate is eliminated by heating at $600^\circ C$.

C. O. C.

Luminescent Material for Use in Fluorescent Paints.

British Thomson-Houston Co.

BP 664,822

Barium zinc silicate of formula $ZnO \cdot BaO \cdot 1.7-12.08SiO_2$ activated with Mn emits a deep red luminescence when exposed to ultraviolet radiation.

C. O. C.

Absorption of Dyes on Chromium Hydroxide (III p. 126).

Development of Coumarone Resins as Raw Material for the Lacquer and Dye Industries (XIII p. 139).

Cation-modified Clays—Plastics, Pigments, etc. (XIII p. 139).

V—PAINTS; ENAMELS; INKS

Manufacture of Liquid Water Colours. H. Hadert, *Farbe u. Lack*, 57, 104 (1951); *Chem. Abs.*, 46, 277 (10 Jan. 1952).

Water colours are made by dissolving unbleached, dewaxed orange shellac (65 g.) in 500 g. of a 1:4 mixture (by volume) of NH_4OH (sp. gr. 0.900) and water on a steam bath, cooling the solution, and extracting it four times with 100 ml. of 50:50 ether and petroleum ether, each extraction taking 3 hr. The ether is removed completely; the shellac content of the solution is determined, about 5 ml. NH_4OH added, water added to bring the solution to 50 g. shellac per 500 ml., and 3 g. borax and 1 g. phenol are added and dissolved by shaking. This basic solution is mixed with an equal volume of aq. dye, and the resulting colour filtered. For 100 ml. water colour the following dye concentrations (g.) are used—Erythrosin Yellow (0.5), Brilliant Orange (0.6), Chloramine Yellow (0.4), Brilliant Green B (1.2), Wool Blue G Extra (0.5), Methyl Violet B (0.5), and Benzamine Brown 3 GO (0.8). These dyes yield very stable and water-resistant colours. Benzo Fast Orange S (0.8), Brilliant Crocine M (0.3), Orange R (0.4), Thiazole Yellow (0.8), Metanil Yellow (0.8), Methylene Blue New N (0.4), Crystal Violet (0.4), and Benzo Brown G (0.6) result in usable though less stable colours. Blacks are obtained by use of lampblack and water-soluble blacks.

C. O. C.

PATENTS

Resinous Ethylene Oxide Derivatives of Phenols for making Lacquers. Ciba.

BP 666,299-300

Combining resinous ethylene oxide derivatives of phenols containing > 1 ethylene oxide group with polybasic carboxylic acids whose carboxyl groups are separated by > 1 C atom and with diacyanamide or a polyamine containing at least 2 NH groups separated by > 1 C atom in presence of an organic solvent containing no free carboxyl group yields heat hardenable lacquers which give films of great hardness, flexibility, and adhesion to either rough or polished metals.

C. O. C.

Inhibiting Sedimentation of Pigments from Paints.

National Research Development Corp.

BP 664,402

Incorporation of a mono- or poly-hydric phenol or a carboxylic acid derivative thereof greatly retards sedimentation of pigments from paints. With many paints, especially those containing a hydrophilic pigment, e.g. ferric oxide, dispersed in a hydrocarbon medium, the effect may be increased by adding in addition an aliphatic alcohol; in such cases the phenol is best dissolved in the alcohol before adding them to the paint.

C. O. C.

Moisture-setting Paint. Sun Chemical Corp.

USP 2,558,665

The vehicle comprises a glycol soluble phenol-formaldehyde resin dissolved in ethylene glycol and/or formaldehyde glycol monoethyl ether, having a water tolerance of up to 45 parts by weight of water per 100 parts of vehicle. When pigmented it yields a paint which rapidly sets at normal R.H.

C. O. C.

Water-emulsifiable Metallic Paste Pigments.

Aluminum Co. of America.

BP 665,709

Water-emulsifiable paste pigments which can be made into paint by mixing with water are obtained by mixing a metallic flake pigment, a hydrocarbon, palmitic acid or stearic acid and/or oleic acid and/or ricinoleic acid, tall oil, a casein binder, and a water-soluble soap-forming alkali.

HP 665,710

C. O. C.

The casein binder is omitted.

Crayon for Marking Rubber. United States Rubber Co.

USP 2,560,195

A crayon made up of wax (600-900 parts), pigment (75-150), and rubber (100) marks rubber permanently but does not interfere with the bonding of the marked surface to another rubber surface to be vulcanized thereto.

C. O. C.

Greenish-blue to Bluish-green Quick-drying Writing Inks. General Aniline.

USP 2,560,881

Copper phthalocyanines of the benzene series containing one or more polyhydroxyalkylphenylphosphonamide groups attached to the phenylene nucleus dissolved in dil. aq. caustic alkali are stable quick-drying writing inks.

C. O. C.

Origins of Pigment and Varnish Techniques (IV p. 129).

Lea-Cyanamide as a Rust-inhibiting Pigment (IV p. 129).

VI—FIBRES; YARNS; FABRICS

Constitution of the Primary Wall of the Cotton Fibre. V. W. Tripp, A. T. Moore, and M. L. Rollins.

Text. Research J., 21, 886-894 (Dec. 1951).

The primary wall of cotton fibres has been isolated by heating in water in a Waring Blender, and then studied by electron microscopic examination and by chemical analyses. It appears to contain about 50% cellulose together with protein, wax, pectic substances, cutin or suberin, and mineral matter. Electron-microscopic examination indicates that the primary wall consists of a network of cellulose fibrils surrounded by the non-cellulosic constituents.

A. B.

Microscopical Observations on the Contents of the Cotton Fibre Lumen. M. S. Catlett, R. Giuffrè, A. T. Moore, and M. L. Rollins.

Text. Research J., 21, 880-886 (Dec. 1951).

The isolation of the lumen contents of cotton fibre by digestion of the cellulose in chilled $72^\circ C$ sulphuric acid is described. The isolated lumens appeared tubular in shape and electron micrographs showed an unoriented, granular structure. The lumen material was shown to be of a proteinaceous nature, and upon acid hydrolysis of the lumen material, amino acids were detected by paper chromatography.

A. B.

Measurement and Physical Interpretation of the Mechanical Strength of Filaments. W. George.

Text. Research J., 21, 847-861 (Dec. 1951).

Evidence for the existence of small domains of size intermediate between atomic and macroscopic dimensions is given. Dimensions from the study of X-ray line broadness as a function of plastic strain seem to correlate with strength properties. A constant true strain rate of fibre deformation mechanism is discussed.

A. B.

Relation between the Strength and Structure of Cotton Fibres. R. A. Dulitskaya and L. I. Belenky.

Tekstil. Prom., 11, 33-34 (Nov. 1951).

The average inclination of the cellulose chain to the fibre axis is determined for each of a wide range of cotton varieties by geometrical and photometric examination of the 002 arcs in the X-ray diffraction pattern. The values are plotted against the tensile breaking stress of the fibre (kg./sq. mm.), and are found to be distributed about a straight line, low chain inclination being associated with

high strength and *rice cerea* (40"—35 kg./sq. mm.; 25"—60 kg./sq. mm.). The line represents the experimental results within $\pm 2\%$. The results confirm the hypothesis that rupture at break is intermolecular. When the different varieties of cotton are examined at just below the breaking point, the differences in orientation are largely leveled out (chain inclination 21–26%). A. E. S.

Heterogeneous Hydrolytic Degradation of Cellulose.

K. Matsuzaki and H. Sobue. *Bull. Chem. Soc. Japan*, **24**, 184–187 (Sept. 1951).

The suggestion that cellulose contains relatively weak bonds at intervals of about 500 glucose units (Husmann, *Makromol. Chem.*, **1**, 140 (1947); **2**, 293 (1948); **4**, 194 (1949)) is criticized. Purified cotton and wood cellulose are hydrolysed with 3.5 N HCl for 4–120 hr. at 30°C., and the hydrolysates nitrated. The resultant nitrates are fractionally precipitated from acetone soln. by means of ligroin, and the degree of polymerisation (D.P.) of each fraction is determined. Integral and differential chain-length distribution curves are plotted, and maxima at varying D.P. are obtained. It is found that wood cellulose gives a less uniform distribution than cotton cellulose, but in each case the fraction with D.P. ca. 500 is insufficient to account for weak bonds. It is considered that the heterogeneous hydrolysis of cellulose may be due to the sub-microscopic structure, and that maxima in D.P. distribution may correspond to multiples of the length of crystalline regions. A. J.

Pyrolysis and Combustion of Cellulose in the presence of Inorganic Salts.

K. Tamaru. *Bull. Chem. Soc. Japan*, **24**, 164–168 (Sept. 1951).

Pyrolysis of cellulose in evacuated quartz tubes produces mainly CO_2 , CO, H_2 , and CH_4 , a residue of carbon (R_1), and in addition unspecified organic vapours which condense and decompose on the wall of the tube producing a solid residue (R_2). The effect of fifty-four inorganic salts on pyrolysis is investigated. In general the amount of R_1 is increased and R_2 decreased. The volume of combustible gases and the velocity of pyrolysis are increased with salts used as fireproofing agents, but the formation of organic vapours (measured as R_2) is inhibited. It is considered that the combustion of cellulose occurs by thermal decomposition followed by ignition of combustible material, particularly organic vapours. A. J.

Polydispersity of Silk.

R. Signer and R. Glanzmann. *Makromol. Chem.*, **5**, 257–261 (1951); *Chem. Abs.*, **46**, 268 (10 Jan. 1952).

Fractional precipitation in conc. aq. Li thiocyanate proves that degummed silk is highly polydisperse and contains molecules with η_{sp}/c varying between 0.8 and 3.0. Silk cannot be degummed without degradation. Fibroin molecules are quickly degraded in cupriethylene-diamine. C. O. C.

Crystal Structure of Silk Fibroin.

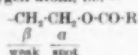
K. Shimura. *Science (Japan)*, **16**, 66–67 (1946); *Chem. Abs.*, **45**, 9982 (25 Nov. 1951).

A new model is proposed having the same size per unit cell as Meyer and Mark's model (*Chem. Abs.*, **23**, 1145 (1929)) and belonging to the monoclinic system. It differs in having alternating polypeptide chains opposite in direction. Every peptide linkage can form hydrogen bonds, and the number of H bonds is far greater than in Meyer's model. This conforms to the theory of Buswell and Ellis (*Chem. Abs.*, **33**, 480 (1939); **34**, 7742 (1940)), based on infrared absorption data, that every peptide linkage forms an H bond. C. O. C.

Thermal Degradation of Polyesters.

H. A. Pohl. *J. Amer. Chem. Soc.*, **73**, 5660–5661 (Dec. 1951).

Thermal degradation of Terylene involves main-chain cracking to smaller molecules. Of a number of polyesters examined the most stable one contained the structure $-(\text{CH}_2\text{CH}_2)_n-$. Introduction of $-\text{COO}-$ groups into this chain, e.g. in sebacic acid-ethylene glycol polyester, considerably reduces the thermal stability. At least one principal point of weakness in the polyesters is in the CH_2 group β to the oxygen atom, i.e.:



This is shown by—(a) replacement of the H atoms on the $\beta\text{—CH}_2$ by CH_3 groups (as in terephthalic acid-2,2-dimethyl-

propane-1,3-diol polyester) increases the stability; (b) introduction of more ether oxygen atoms (as in terephthalic acid-diethylene glycol polyester) decreases the thermal stability. C. O. C.

Preparation of Perlon from ϵ -Caprolactam.

A. Mathies. *Makromol. Chem.*, **5**, 197–244 (1951); *Chem. Abs.*, **46**, 268 (10 Jan. 1952).

Formation of Perlon from ϵ -caprolactam in presence of acid catalyst leads to equilibrium between the amount of polymer, lactam, degree of polymerisation, and catalyst. Growth of chains is compensated by two different polymerisation reactions—(a) fission of chains by the catalyst, and (b) thermal splitting of lactam from the ends of the chain. The formation seems to be an addition of radicals. C. O. C.

Flow through Fabric-like Structures.

S. E. Panner and A. F. Robertson. *Text. Research J.*, **21**, 775–788 (Nov. 1951).

A visual investigation of flow through fabrics is made by forcing a liquid past models representing the four possible combinations of yarns in a woven fabric interstice, the fluid motion being visualised by using air bubbles as indicator. It is shown that the four pore types do not behave as geometrically similar orifices, the effective pore area decreasing with increasing Reynolds number. A. B.

PATENTS

Drying Long Fibres.

J. H. Zimmermann. *BP* 665,605. Fibres such as sisal, hemp, jute, and ramie are arranged in a layer with the thick ends aligned, and are gripped near the thinner ends by a gripper which can be hung on a rack, or transport chains, with the thick ends hanging downwards, and are thus hung in or passed through a drying chamber. The layers may be so shaped that they can be conveniently drawn into a press box for baling. J. W. B.

Stabilisation of Polyacrylonitrile.

DuPont. *USP* 2,560,653. Addition of 0.1–2.0% (by weight) of sulphuric acid, an organic sulphonic acid, or a hydrated salt of an organic sulphonic acid, together with the same weight of a thiol, to a solution of an acrylonitrile polymer (containing at least 85% by weight of acrylonitrile) prevents discoloration of the solution when it is heated or kept. Fibres spun from such solutions can be dyed much brighter than can fibres spun from solutions not containing these stabilisers. C. O. C.

Acrylonitrile Copolymers.

American Viscose Co. *USP* 2,560,680. Acrylonitrile-acrylamide copolymers containing 70–95 mole % of acrylonitrile because of their plasticity are especially suitable for forming into filaments. C. O. C.

Coloured Polyacrylonitrile Fibres.

American Cyanamid Co. *USP* 2,558,735. Polyacrylonitrile dissolved in a conc. aq. soln. of a water-soluble salt which yields highly hydrated ions, e.g. a thiocyanate, is extruded into an aqueous coagulating bath at not $> 10^\circ\text{C}$. to produce a fibrous gel which can be oriented by wet stretching. In this gel state the fibres are much more readily dyed, e.g. in an aq. dispersion of 4- β -hydroxy-ethylamino-1-methylaminoanthraquinone, 4-acetamido-2'-hydroxy-5'-methylazobenzene, or 4-bis- β -hydroxy-ethylamino-2-methyl-4'-nitroazobenzene, than they are after drying, which destroys the gel structure. C. O. C.

The precipitated water-swollen gel is wet-stretched at 70–110°C. and then treated with a 10–80% aq. soln. of urea or a non-polymerisable alkyl- or hydroxyalkyl-urea. This prolongs the gel life of the precipitated product. C. O. C.

Aqueous Colloidal Dispersions of Polytetrafluoroethylene for Extrusion as Filaments or Films.

DuPont. *USP* 2,559,750. Filaments, films, etc. may be formed by extruding a not $< 5\%$ aqueous colloidal dispersion of polytetrafluoroethylene, in which all the particles have at least one dimension $< 0.1 \mu$, and at least 5% have an elongated, ribbon-like shape of width $< 0.07 \mu$ and a length: width ratio $> 5:1$, and then sintering the formed filament, etc. at $> 327^\circ\text{C}$. C. O. C.

Aqueous Colloidal Dispersions of Polymers.

DuPont. *USP* 2,559,752. Unsaturated organic compounds are formed into aqueous dispersions with the aid of compounds of formula

$B-(CF_3)_n(CH_2)_m-A$ ($B = H$ or F ; $n > 4$; $m = 0$ or 1 ; $m + n > 5$; $A =$ an ionic hydrophilic group) and then polymerised. The resulting dispersions, e.g. of tetrafluoroethylene, are suitable for spinning into filaments, for coating or impregnating textiles and paper, etc. C. O. C.

N-Substitution in Polyamides (XIII p. 138).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

PATENTS

Compressional Wave Energy as an Aid in Washing. Lever Brothers & Unilever. BP 664,502

The goods to be cleaned are treated with a cleansing liquid which is subjected to compressional wave energy, e.g. supersonic vibration, the goods being moved about relative to the nodes and anti-nodes of the standing wave formed by the energy. Uniform cleansing is obtained. C. O. C.

Bleaching with Chlorite. Solvay & Cie. BP 665,265
Chlorine dioxide is evolved only within or at the surface of the fibres if they are treated with an activating agent, e.g. a compound of an element of group V or VI (cf. BP 639,235—J.S.D.C., 66, 496 (1950)) or a hydrolysable organic solvent (cf. BP 651,405—J.S.D.C., 67, 288 (1951)), before being entered into the chlorite bath. C. O. C.

VIII—DYEING

Theory of the Dyeing of Cellulose Fibres with Direct Dyes. F. I. Sadov and K. G. Kalina. Tekstil. Prom., 11, 36-38 (Nov. 1951).

A study is made of the direct dyeing of cotton and of products of the selective oxidation of cotton, viz. monocarboxycellulose preparations (COOH cont. at 0.4-65% of theoretical max.) obtained by oxidation with N_2O_4 ($-CH_2OH \rightarrow -COOH$) and dialdehydocellulose preparations (CHO content 0.4-11% of theoretical max.) obtained by oxidation with periodic acid ($-CHOH-CHOH- \rightarrow -CHO + OHC-$). Dyeing is done at 55°C. and a liquor ratio of 200:1 with purified Direct Pure Blue (C.I. 518) (0.03 g./litre) for 1 hr. and for 48 hr. In presence of 0.3 g. sodium chloride per litre, the dialdehydocellulose preparations absorb more dye than the original cotton, the effect being greatest in the preparations of low CHO content and falling off as the degree of oxidation increases. The monocarboxycellulose preparations do not dye, but as the pH of the dyebath is 2-3.5, it is surmised that the electrokinetic potential is high. Increase in salt concn. to 3-100 g./litre leads to absorption of dye in amounts that diminish with increasing COOH content. A preliminary treatment of the monocarboxycellulose with dil. calcium acetate soln. gives a product that dyes readily, the dyebath being now neutral. Nylon, which differs from cellulose in the absence of the primary alcohol side-chain, does not dye under the conditions used for cellulose. A. E. S.

Azoic Dyeing of Wool. B. M. Bogoslovsky, P. G. Levinsky, and M. V. Pavlovskaya. Tekstil. Prom., 11, 38-39 (Nov. 1951).

Fast dyes on wool are obtained by dyeing with an ammoniaphthosulphonic acid, which is then coupled with a diazotised amine. The pH of the coupling bath should be 6.3-8.3. In particular, H acid coupled with Variamine Blue B gives a blue dyeing that is converted by dichromate into a very fast brown. A large-scale trial with this method of dyeing is described. Loose wool (105 kg.) was dyed with H acid (2%) in presence of sulphuric acid (2%) (liquor ratio 30:1, 10 min. warming up, 20 min. at boil), rinsed, treated with Variamine Blue B Salt (4%, 30 min.), again rinsed, washed with hot HCl, and finally with cold water. The total processing time was 2 hr. For comparison, another batch of the wool was dyed with a dye of the Coomassie Navy Blue type; in the subsequent combing and spinning, the azoic-dyed wool behaved in the more satisfactory manner. After weaving, the azoic-dyed wool was chromed (6% dichromate, 6% sulphuric acid, liquor ratio 15:1, ~90°C., 20 min.) to give the brown dyeing. The subsequent processes, which included heavy milling in soap and soda and carbonising, are described. The finished cloth was up to standard in all respects. A. E. S.

Sorption of Acid Dyes by Nylon under Non-equilibrium Conditions. F. C. McGrew and W. H. Sharkey. Text. Research J., 21, 875-879 (Dec. 1951).

The unevenness in acid-dyed nylon due to differences in orientation and in the number of amine end groups has been shown to be minimised by dyeing in a very dilute dyebath. Systems by which dye is supplied to the fibre at a rate below that at which it is sorbed were found effective, e.g. by application of the dye to nylon by transfer from wool, by controlled mechanical addition of dye to the bath, and by use of the dye in the form of a slightly soluble heavy-metal salt. A. B.

Unified Picture of Diffusion. J. D. Babbitt. Canadian J. Physics, 29, 427-436 (Sept. 1951).

Starting with the fundamental equation—

$$\frac{dp}{dx} = A_x$$

(p = a pressure function at a point x ; A_x = a resistive or frictional force), the specific equations are deduced for diffusion in solution in terms of concentration gradient, chemical potential, or mobilities. The interdiffusion of ideal gases is shown to be expressed by Fick's law and its application is extended to non-ideal gases. Other treatments include the diffusion of metals in metals, and gases through adsorbing solids. By these means a unified theoretical picture of diffusion is presented. L. P.

Diffusion of Adsorbed Gases through Solids. J. D. Babbitt. Canadian J. Physics, 29, 437-446 (Sept. 1951).

The above theory is applied to a diffusion process consisting in the spreading of a monolayer. Mobile monolayers, and localised ones obeying the Langmuir or Brunauer-Emmett-Teller isotherm, are considered, and the equations are compared with the experimental data for the diffusion of hydrogen through copper and that of water vapour through keratin. A graph is given which shows the theoretically expected variation of the diffusion coefficient with vapour pressure for a material whose adsorption isotherm follows the B.E.T. equations. L. P.

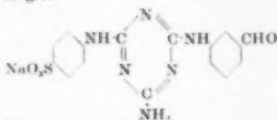
PATENTS

Dyeing by the Molten Metal Process. Standfast Dyers & Printers. BP 665,454

The layer of dye liquor on top of the molten metal is maintained at comparatively small volume by a continuous supply of fresh liquor. The replenishing liquor is heated as it is supplied to this layer. C. O. C.

Water-soluble Aldehydes with Affinity for Wool—Dyebath Assistants for Metalliferous Azo Dyes. Ciba. BP 661,912

When dyeing certain metallised o-hydroxy-o'-aminomonazo dyes, the presence of water-soluble aldehydes having affinity for wool inhibits decomposition of the dyes and results in brighter dyeings. Aldehydes containing the cyanuric ring are particularly valuable, but coloured (especially yellow) compounds, e.g. the monazo compound *m*-aminobenzaldehyde-1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone, are also useful. Thus, cyanuric chloride is condensed first with 1 mol. of sulphonic acid and then with 1 mol. of *m*-aminobenzaldehyde in presence of soda ash, and finally heated with ammonia to give—



This compound is added to the dyebath to the extent of 1-2% on the weight of wool when dyeing with 1-5% of the chromium complex of the monazo dye 5-nitro-2-aminophenol-2-naphthylamine-6-sulphonic acid, when a pure green dyeing results. Without the soluble aldehyde addition, bluer and duller dyeings are obtained. E. S.

Colouring Textiles with Pigment Dispersions. IC.

USP 2,558,033

Dyeings or prints of good fastness to rubbing are obtained by using a pigmented dispersion of a normally

solid high-mol.wt. polymer of ethylene in a volatile organic liquid. Preferably the composition also contains a thermosetting resin soluble in the solvent used. C. O. C.

Dyeable Corrosion-resistant Coating on Zinc and Zinc Alloys. Plasco. BP 665,993

The metal is treated with an aq. soln. of a Cr_2O_3 -containing compound, e.g. sodium dichromate, lactic acid and/or a soluble lactate, and a strong mineral acid at pH 0.65-1.5. The treated metal while still wet is washed, dried, and baked to fix the dyed surface film. C. O. C.

Solubilised Azoic Dyes (IV p. 129).

Azo Printing and Dyeing (IX this page).

IX—PRINTING

Production of Handkerchiefs. M. Braun-Ronsdorf. *Ciba Review*, No. 89, 3213-3218 (Dec. 1951).

Brief account of the history of the printing of handkerchiefs. C. O. C.

Chromatographic Investigations related to Photographic Theory. T. H. James and W. Vanselow. *J. Amer. Chem. Soc.*, 73, 5617-5622 (Dec. 1951).

A series of photographic sensitising and densitising dyes were separated by elution chromatography using AgBr as the adsorption medium and aqueous pyridine as the chromatographic developer. The following water-soluble dyes were used to determine relative adsorption of various colourless compounds by AgBr—phenosafranin, 3,3'-diethyloxycarbocyanine chloride, Methylene Blue, 3,3'-diethylthiacarbocyanine *p*-toluenesulphonate, and 1,1'-diethyl-2,2'-carbocyanine chloride. The ease of displacement of the dyes from the AgBr column decreases in the order given. The chemical sensitiser, allylthiourea, displaces all five dyes with about equal ease and is more strongly adsorbed than any of them. The photographic developers, hydrazine and *p*-phenylenediamine, displace phenosafranin and 3,3'-diethyloxycarbocyanine, showing that they themselves are adsorbed. Quinol and catechol in aqueous solution at pH 8 mordant the dyes, showing that developers are adsorbed in presence of the dyes. Gelatin displaces phenosafranin and 3,3'-diethyloxycarbocyanine but not the other dyes. C. O. C.

PATENTS

Azo Printing and Dyeing. General Aniline.

BP 665,587

Variations in hue when using metallised *o*-carboxy- and *o*-hydroxy-arylazoresorcinols in continuous dyeing or printing are avoided if, after coupling is completed, the material is treated with a mineral acid to remove alkali metal bound by the dye on the fibre. C. O. C.

Printing with Dispersed Dyes. ICI. BP 664,646

Much brighter prints are obtained with dispersed water-insoluble dyes if the printing paste contains a condensate of ricinoleic acid and/or bodied ricinoleic acid with ethylene oxide. C. O. C.

Electrostatic Printing. W. C. Huebner.

USP 2,558,900-1

Printing on Polyvinyl Chloride. Decora Corp.

USP 2,558,791

An ink containing polyvinyl chloride is printed on to a non-elastic transfer sheet. The printed surface of the transfer sheet is then pressed on to the polyvinyl chloride sheet after both have been heated to a temperature sufficient to soften the ink and cause it to adhere to the polyvinyl chloride sheet. C. O. C.

Drying of Steam-setting Printing Inks. A. F. Schmutzler.

USP 2,558,787

Raising the moisture content of the paper before printing results in more rapid hardening of prints from steam-setting inks on drying. C. O. C.

Typewriter Ribbon. National Cash Register Co.

BP 664,456

The ribbon is impregnated with a substance, e.g. 3,3-bis-*p*-dimethylaminophenyl-6-dimethylaminophthalide, dissolved in a non-evaporating liquid, which substance when transferred to paper, etc. reacts with a second substance present in the paper, e.g. barium sulphate, to form a coloured impression. C. O. C.

Decalcomania (Transfer) Paper. McLaurin-Jones Co. USP 2,561,976

Firm bonding is produced between the decalcomania print and the surface to which it is to be applied by using a mixture of highly concentrated rubber latex and water-soluble adhesives, this mixture being such that when applied to the paper backing the latex globules remain water-sensitive after the coating has been dried. C. O. C.

Hardening Dye-absorptive Hydrophilic Colloidal Layers. Technicolor Motion Picture Corp.

BP 664,597

Gelatin or other dye-absorptive hydrophilic colloidal layer of a blank film is hardened preparatory to dye transfer by treating it with an aqueous solution of a chromic salt and then rinsing. The solution contains 25-70% of the green form of the salt and 75-30% of the purple form. The purple form is the hardening agent, while the green form acts solely as a mordant for the dye. C. O. C.

Photographic Imbibition Dye Printing. Kodak. BP 664,836

Images of controlled contrast are obtained by treating an imbibition dye printing matrix with an imbibition dye-bath and a water-soluble colourless arylsulphonic acid, and transferring by imbibition a dye image from the dyed matrix to a printing blank. C. O. C.

Thallium Salts in Photographic Colour Developers. General Aniline. USP 2,561,166

Addition of soluble thallium salts to the colour developer for colour development of multilayer photographic colour emulsions of the negative and reversal type materially improves the developing characteristics and prolongs the life of the developer. C. O. C.

Colour Photography. Kodak. BP 666,107

Developing an exposed layer containing AgBr and AgCl with a primary aromatic amino developing agent containing as antioxidant 6-nitrobenzimidazole or its nitrate, 5-nitroimidazole, or benzotriazole enables the AgCl to be individually developed. C. O. C.

Colour Photography. Ilford. BP 665,180

Colour Photography. K. O. Ganguin. BP 665,349

Colour Photography. DuP. BP 665,657

Transfers for Ceramics. Meyercood Co. BP 664,837

Solubilised Azoic Dyes (IV p. 129).

Colouring Textiles with Pigment Dispersions (VIII p. 134).

X—SIZING AND FINISHING

Sizing without Fat. A. V. Kuligin. *Tekstil. Prom.*, 11, 37-39; M. F. Smirnov. *Ibid.*, 11, 40 (July 1951).

Experience of many weavers shows that in the sizing of cotton warps with starches (and also rye and wheat flour) modified by the chloramine method (see J.S.D.C., 66, 457 (Aug. 1950)), the addition of fatty substances or of glycerol is unnecessary. A. E. 8.

Chemical Substitution in Fibrous Cotton and Resistance of Substituted Cotton to Microbiological Deterioration. C. F. Goldthwait, E. M. Burns, and A. S. Cooper. *Text. Research J.*, 21, 831-840 (Nov. 1951).

The acetylation of cellulose is shown by dyeing tests, involving the use of selective dyes for cellulose and acetylated cellulose, to take place along fibres in various segments and throughout cross-sections rather than as a layer increasing in thickness from the surface. At one-third substitution the amorphous cellulose is converted to triacetate. Protection from micro-organisms is believed to be due to the blocking of hydroxyl groups, the filling of spaces in amorphous cellulose, and the suppression of swelling. A. B.

Fundamentals of Flame Retardancy. R. W. Little. *Text. Research J.*, 21, 901-908 (Dec. 1951).

The mechanism of the combustion of a textile fabric is discussed. The principal effect of an efficient flame retardant appears to be reduction in the amount of combustible tarry products and an increase in the amount of

solid char formed. The mechanism of glowing combustion and the effect of glow inhibitors are also discussed.

Felting of Wool as related to the Elastic and Swelling Behaviour of the Fibre. H. Bogaty, A. M. Sookne, and M. Harris. *Text. Research J.*, **21**, 822-826 (Nov. 1951).

A study of the felting of wool top in a variety of organic and inorganic reagents is described. The feltability is shown to vary approximately directly with the fibre swelling in each reagent and inversely with the work of extension of the wet fibre. It is also shown that there is a close linear relationship between the felting shrinkage of wool top and the resilience of the single fibre in the wet state, enabling an estimation of feltability to be made from the single fibre properties. Fibres dyed to saturation with Orange II exhibit elastic behaviour similar to that for undyed fibres in a poor felting medium.

Felting of Shrink-resistant Wool as related to Some Properties of the Single Fibre. A. M. Sookne, H. Bogaty, and M. Harris. *Text. Research J.*, **21**, 827-830 (Nov. 1951).

The feltability of wool treated to various degrees of shrink-resistance by a wet chlorination process has been determined in solutions ranging from very good to very poor felting promoters. The shrinkage behaviour has been shown to be related in a simple equation to the fibre resilience and to the fibre frictional properties measured in the same reagents.

Finishing Fabrics containing Rayon Staple Fibre. F. I. Raykhlin and N. M. Kasatkina. *Tekstil. Prom.*, **11**, 24-26 (July 1951).

A description is given of the use of a new finishing agent, Preparation AMD, on mixture fabrics containing wool and viscose or cuprammonium staple fibre. The agent contains hydroxymethylurea and the *N*-hydroxymethylamides of higher fatty acids. It is applied by padding in presence of lactic acid, drying, and then curing at high temp. It confers water-repellency, dimensional stability, increased wet strength, and improved handle. The finish is fast to light and dry-cleaning, and is moderately fast to washing.

Impregnation of Fabrics with Synthetic Latexes. Ya. M. Yabko and S. S. Voyutsky. *Tekstil. Prom.*, **11**, 26-27 (July 1951).

Cotton fabric is impregnated with various synthetic rubber latexes. The uptake shows no clear relation to the pH or viscosity of the latex. It is correlated poorly with the surface tension γ of the latex, but shows a close correlation with the "wetting" (cosine of contact angle θ) and with the "work of spreading", $\gamma(\cos \theta - 1)$. High uptake is associated also with low sorption of the dispersed phase on to the fibre.

PATENTS

Improving the Action of Caustic Lye on Cellulosic Textiles while avoiding Harmful Secondary Effects. Solvay & Cie. *BP* 664,914

Adding a small amount of a water-soluble chloride (e.g. 0.2-5.0 g./litre) to the lye allows more vigorous mercerising while preventing harmful secondary reactions.

Chemical Modification of Cellulosic Materials to impart Dimensional Stability and Crease Resistance. United States Rubber Co. *BP* 664,795

The material is treated with aq. vinyl sulphone and then with an aq. alkaline catalyst. Depending primarily upon the strength and concentration of the catalyst used, the reaction is complete in a few seconds to 2 hr. The temperature used is preferably < 50°C.

Crease-resisting of Cellulosic Textiles. American Cyanamid Co. *BP* 664,993

The material is impregnated with (a) a curable hydroxymethylmelamine which may be alkylated, and (b) a film-forming, water-insoluble thermoplastic alkyl acrylate polymer or copolymer with a monomer containing a $\text{CH}_2\text{C}=\text{C}$ group. The proportion of a : b by weight is 1 : 1-3, and the amount of the two retained by the material after curing and washing is 8-40%, on its dry weight. The process is particularly applicable to mercerized cotton fabrics.

Lustrous Titanium-impregnated Cellulosic Materials of Reduced Flammability. DuPont, *USP* 2,563,656

Regenerated cellulose is impregnated with a water-soluble Ti compound, then rinsed in an aqueous solution of a sulphate, nitrate or chloride, and finally treated with a compound which precipitates a water-insoluble Ti compound in the fibres. The treated fibres must contain 3-20% by weight of the insoluble Ti compound.

Bonding Viscose Rayon and Rubber. Dunlop Rubber Co. *BP* 665,619

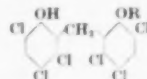
Better adhesion is obtained if the rayon is first coated with casein.

Saponification of Cellulose Esters in Packages. Celanese Corp. of America. *BP* 664,040

The time required to saponify cellulose esters, e.g. a package containing cellulose acetate yarn, is considerably reduced by interrupting the flow of the saponifying agent through the package at intervals.

Rendering Cellulose Bacteriostatic. Pacific Mills. *USP* 2,559,986

Chemical reaction of cellulose with a compound of formula—



(R = $-\text{CH}_2\text{CH}(\text{OH})\text{CH}_2\text{OCH}_2\text{Cl}$ or $-\text{CH}_2\text{CH}(\text{OCH}_2\text{Cl})\text{CH}_2\text{OCH}_2\text{Cl}$),

e.g. by heating them together in presence of sodium acetate, renders it bacteriostatic. Its affinity for water is also increased, but without causing it to reach a degree of solubility undesirable in textiles.

Strengthening Regenerated Protein Fibres. ICI. *BP* 665,462

The insolubilised filaments are treated after acid dyeing, and preferably under tension, with an aq. soln. of a salt of Hg, U, Cr, Zn, Cd, or Al.

Inhibiting Gas Fading. BrC. *BP* 665,401-2

A water-insoluble nitrogenous organic gas fading inhibitor either in aqueous emulsion or dissolved in a water-insoluble solvent is applied to a coloured cellulose ester or other to impart a finish which is fast to dry cleaning or washing. *NN'*-Dibenzylethylenediamine is especially suitable for application by this method, 0.3-1.0% by weight being impregnated into the fibre.

Delustering Polyester Fibres. Calico Printers' Association. *BP* 664,921

Treatment with 30-50% aq. NaOH or KOH at 15-65°C. is used.

Sizing Nylon. Monsanto. *BP* 665,199

Nylon warps are sized with an aq. soln. of an ammonium, quaternary ammonium, Na, or K salt of a styrene- or subst. styrene-alkyl (C_{1-4}) maleate copolymer in absence of substances which when heated with the copolymer would form an insoluble resin. Such a size is easily removed by warm water.

Setting Tricot Fabrics. American Viscose Corp. *BP* 664,612

The fabric is uncured and then fed through a heating zone, e.g. an aqueous bath, under no transverse tension and only such longitudinal tension as is necessary to pass it through the heating zone, guiding members being provided to prevent the fabric from curling in the heating zone.

Imparting Dimensional Stability to Thermoplastic Fabrics. J. Cadene. *USP* 2,563,394

Nylon, acetate rayon, etc. fabrics are passed under tension in both length and width through molten metal at a temperature sufficient to plasticize the fibres. While in the molten metal the fabrics pass through squeeze rollers to ensure that the molten metal touches the entire surfaces of the yarns composing the fabric.

Treatment of Polyurethane Fibres with Formaldehyde. BrC. *BP* 664,182

The softening point and textile properties of polyurethane fibres are improved by treating with formaldehyde in alcoholic solution at pH > 10.

Water-repellent Finish. Socony-Vacuum Oil Co.

USP 2,559,260

A water-repellent finish fast to dry cleaning and washing is obtained by impregnating fibrous material with an aqueous solution of a water-soluble halide of a nitrogenous condensate containing at least one 5-membered heterocyclic nucleus (obtained by condensing thiophen, furan, or pyrrole with formaldehyde and an ammonium or hydroxylamine halide in presence of an acid of ionisation constant 1×10^{-3} to 1.7×10^{-2}). It is then treated with alkali, if desired rinsed, and then treated with formaldehyde, baked at 212–260° F., and rinsed. C. O. C.

Improved Bond between Glass Fibres and Resins.

Owens-Corning Fiberglass Corp. USP 2,563,288

Better bonding between glass fibres and plastics or resins is obtained if the fibres are first treated with a compound of formula $(R^1-CH_2-CR^2-CH_2)_n-SiX_{4-n}$ ($n = 1-3$; R^1 and $R^2 = H, Alk, Ar, Cl, Br, or I$; $X = Hal, alkoxy, or aryloxy$), e.g. allyltriethoxysilane, or a hydrolysis or polymerisation product of such a compound. USP 2,563,289

Strong bonding is obtained even at high humidities if the fibres are first treated with an amino polymer containing an $>NR^1R^2X$ group (R^1 and $R^2 = H, Alk, Ar, aralkyl, or heterocyclic$; $X = a$ highly ionisable anion), e.g. polyorthovinylpyridine. C. O. C.

Rendering Siliceous Surfaces Organophilic. Corning Glass Works. USP 2,557,786

Siliceous surfaces, e.g. glass, become organophilic when treated with organosilicon halides. Organic resins adhere much better to such treated surfaces than to the untreated. C. O. C.

Transfer Coating. American Viscose Corp.

USP 2,559,649

Textiles, paper, etc. are brought into contact under heat and pressure with a movable support carrying a thin thermoplastic film. They are then cooled before stripping them and the film which adheres to them from the support at a point removed from where the heat and pressure were applied. C. O. C.

Pyrolysis and Combustion of Cellulose in the presence of

Inorganic Salts (VI p. 133).

Drying Long Fibres (VI p. 133).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS**Production of Wood Cellulose by the aid of Organic Solvents.** V. V. Yanovsky and S. S. Malevskaya.

J. Appl. Chem. U.S.S.R., 24, 1100–1108 (Oct. 1951).

Satisfactory delignification of deciduous wood (asp and birch), but not of pine and fir, was obtained by boiling under pressure (4–9 atm.) with a mixture of equal parts of water and organic solvent (glycol, butyl alcohol, amyl alcohol, or glycol monoethyl ether). The best results were obtained with glycol monoethyl ether. After bleaching, the fibre obtained (81–86% α -cellulose) was of good mechanical properties. A. E. 8.

Solutions of Cellulose Acetates of Varying Acetyl Number. A. B. Pakhshv and G. K. Kopylova.

J. Appl. Chem. U.S.S.R., 24, 1052–1057 (Oct. 1951).

Specific viscosities are determined in various solvents of cellulose acetate preparations of const. mol. wt. but of varying acetyl number (49.9–61.4). The solvents used are: acetone, acetone-ethanol, aqueous formic acid, dichloroethane-ethanol, and methylene dichloride. It is shown how the results can be interpreted qualitatively in terms of the interaction of groups in the polymer amongst themselves (high viscosity) and with solvent or non-solvent molecules (low viscosity). A. E. 8.

Vinylation of Cellulose. V. V. Shtshkevsky, N. A.

Obolonskaya, and N. I. Nikitin. J. Appl. Chem. U.S.S.R., 24, 1045–1051 (Oct. 1951).

An attempt is made to vinylate cellulose by treatment with acetylene at 130–175° C. for 18–75 hr. in presence of a 20–40% soln. of KOH. The main product is insoluble fibrous material, of composition $2C_6H_{10}O_2 \cdot C_2H_2$ or $2C_6H_{10}O_2 \cdot 3C_2H_2$ according to conditions. On hydrolysis with acid it gives considerably less acetaldehyde than would be expected for a vinyl ester; also, it does not combine with bromine. It is suggested that vinyl groups are

introduced into the cellulose molecule and then react with OH groups in the same or neighbouring chains, forming bridges of the type $-O-CH_2(CH_2)_2-O-$. A. E. 8.

PATENTS

Dimensionally Stable Paper. J. T. Stearns.

USP 2,559,234

Formation of a thiourea-formaldehyde condensate in the fibres followed by heat curing yields paper which is dimensionally stable under changing moisture conditions. C. O. C.

Urea-Formaldehyde Compositions for producing Paper of High Wet Strength. DuP. USP 2,557,299

High wet strength is imparted to paper by treating it with the product obtained by condensing urea with formaldehyde and with 2–40 parts by weight of diglycolic acid per 100 parts of urea and formaldehyde. This condensate has high affinity for cellulose, and can be applied either in the beater, in tub-sizing, or by being sprayed into the paper. C. O. C.

Paper of Improved Wet Strength. American Cyanamid Co. USP 2,559,220

Positively charged melamine-aldehyde resin added as a colloidal solution to paper stock exhausts on to the fibres. USP 2,559,221

Controlling the sulphate ion in the water within 40–150 p.p.m. increases the degree of exhaustion of the resin on to the fibres. C. O. C.

Soya-bean Flour Coating for Paper. Watervliet Paper Co. USP 2,559,901

Smooth paper of improved ink receptivity is produced by use of a coating prepared by treating soya-bean flour with an amylolytic enzyme and an alkali and mixing the solubilised flour with an earthy filler. C. O. C.

Coating Paper on the Fourdrinier Machine. West Virginia Pulp & Paper Co. USP 2,560,572

Rubber-Lignin Composition for Impregnating Paper. DuP. USP 2,558,634

Paper is made more waterproof, stiff, hard, and resistant to abrasion by impregnation with an aqueous rubber-lignin dispersion stabilised with an ammonium compound. C. O. C.

Paper of Stabilised Water Content. A.S.P. Chemical Co. BP 664,848

In order to stabilise the inherent water content of the finished sheet, to lessen the amount of size, etc. required to impart gloss, and to prevent the creeping of ink, water-stabilising materials in the form of comminuted vegetable plant flesh, including the external coverings of plant growth, are added to the raw paper pulp with alkali reagents to form alkali pectates *in situ*. A prepared alkali pectate, e.g. sodium or ammonium pectate, may be added as a sol or a gel, with a proportion of vegetable coverings. The sol may be converted into a gel by adding a metallic salt, e.g. copper, zinc, or aluminium chloride, or by acidifying the pulp with an inorganic or vegetable acid, e.g. citric pulp. The vegetable coverings may be added simultaneously with the plant flesh and alkali or the alternative pectic sol or gel, and the pectic gel and vegetable matter and coverings may be added as a dry powder. S. V. 8.

Treatment of Paper with Liquids during its Manufacture. E. G. J. Froehlich. BP 664,239

Sizing materials, e.g. natural and artificial resins, vegetable, animal and mineral waxes, bitumens, distillation residues (e.g. petroleum, asphalt), montan wax or stearic acid, artificial waxes, fats, etc. dissolved in mineral oil fractions of sp. gr. 0.70–0.90, are introduced on the paper machine, after the last press, into the otherwise completed structure of the still moist paper. The sizing solution may be fed to the paper only at predetermined places, to produce shadow watermarks, etc. S. V. 8.

Coated Paper. North America Paper Process Co.

BP 664,949

A discontinuous coating of fine uniform pattern is applied to paper, etc. by passing the web between a backing and a coating roll, the latter having minute depressions formed in a uniform pattern in the surface from which the coating is deposited. The coating is then solidified, and finally pressed into the sheet. The coating may be applied by direct printing and solidified by dehumidifying. The individual coated and uncoated areas are so small that, after calendering to level the coating, they are not readily

independently distinguishable at normal distances for vision. S. V. S.

Multicoloured Carbon Paper. J. R. Ehrlich.

USP 2,559,608

The ink used contains an insoluble pigment (except a black) and a soluble dye of a different colour. Sufficient of the dye should be present either to produce together with the pigment a different ink colour or to cover completely the colour of the pigment. The dye used must be capable of being either discharged or altered in colour by agents which do not affect the pigment. This enables chemical treatment of copy from the paper to make the colour of the pigment appear either dominant or modified by the changed colour of the dye, thus causing the treated portion to assume a contrasting colour. C. O. C.

Organic Triphosphites as Stabilisers for Cellulose Ether Compositions. Brit. BP 664,549

An organic triphosphite (3% or more) stabilises cellulose ether compositions to heat and light. C. O. C.

Stabilisation of Cellulose Ethers. Hercules Powder Co. USP 2,561,892.3

Copper and copper compounds stabilise cellulose ethers or their compositions to heat and oxygen. C. O. C.

Transfer Coating (X p. 137).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Pliability in the Dressing of Fur Skins. H. Erdmann and U. H. Dienstmann. *Die Leder*, 2, 173-189 (1951); *Chem. Abs.*, 45, 10,633 (25 Nov. 1951).

An apparatus which measures stretch and recovery of the skin under increasing loads is described. The pliability of rabbit skins was not appreciably increased by addition of salt or formic acid to the soak liquors. There was direct relationship between acid concentration of the pickle, loss of protein, and pliability. Skins tanned with Cr or H-CHO were pliable enough when first given an H_2SO_4 pickle. C. O. C.

PATENTS

Drying Fur-bearing Animal Skins. Jonas & Saumburg Corp. BP 665,162

Carrotting solution is applied to fur-bearing skins, which are then passed through a drying zone of such radiant energy that the fibres would singe if stationary; a current of air is blown over in the same direction. Conditions are described whereby less solution is required, intermediate conditioning between carrotting and drying is eliminated, and improved colour and felting properties result.

J. W. B.

Protecting Furs from Biological Attack. E. B. Higgins. BP 666,189

The fur is drummed with a powder moistened with a solution of pentachlorophenyl laurate, e.g. sawdust and a white-spirit solution of the laurate. Both hair and hide are penetrated. C. O. C.

XIII—RUBBER; RESINS; PLASTICS

Quantitative X-Ray Studies of Order in Amorphous and Crystalline Polymers—Scattering from Various Polymers and a Study of the Glass Transition in Polystyrene and Polymethyl Methacrylate. S. Krimm and A. V. Tobolsky. *Text. Research J.*, 21, 805-822 (Nov. 1951).

The application of a Geiger counter spectrometer to the quantitative study of the scattering from various amorphous and crystalline polymers has been investigated. The scattering patterns are analysed for their structural implications. It has been found that the X-ray diffraction patterns of polystyrene and polymethyl methacrylate change as these materials are heated through the region of their second-order transition point, these changes being interpretable in terms of definite changes in structure of the polymeric chains. A. B.

Urea-Formaldehyde Condensates. G. Zigeuner. *Kunststoffe*, 41, 221-224 (1951); *Chem. Abs.*, 45, 10,662 (25 Nov. 1951).

An investigation of the urea-formaldehyde condensation, especially of the problem of formation of ureido-

methyl ethers, which is important in alkaline and probably also in weakly acid condensation. The derivatives of N-(hydroxymethyl)amides (I) and their structural changes are described. Their reactivity in acid or alkaline solution is due to the polarisability of the CH_2-OH bond, which makes possible the anionoid separation of the O atom in the CH_2OH group or the N atom of a second molecule of a CH_2OH compound, the pH controlling the course of the reaction. In alkaline media and possibly also at pH 4-7 breaking of the CH_2-OH bond by the O atom in the CH_2OH group of a second molecule of a CH_2OH compound (formation of bis(acylaminoethyl) ethers and ureido-methyl ethers) is preferred, while in presence of mineral acids (pH 4) the N atom of a second molecule of a CH_2OH compound causes breaking of the polarised CH_2-OH bond. Simultaneously the CH_2OH group on this N atom is displaced with formation of $H-CHO, H_2O$ and the corresponding methylene compound. The CH_2OH compounds of the ureas are more reactive than I. Twenty-two different structural diagrams of derivatives and 17 references are given. C. O. C.

Urea-Formaldehyde Reaction—I, II, and III. A. Takahashi. *Chem. High Polymers* (Japan), 7, 115-121, 165-169, 169-177 (1950); *Chem. Abs.*, 46, 438 (25 Jan. 1952).

I—When urea reacts with formaldehyde in neutral or alkaline media the pH of the system decreases as the reaction proceeds. When ammonia or hexamethylenetetramine is used as catalyst, the pH increases at first to a maximum and then decreases, but with NaOH or $Ba(OH)_2$ it decreases immediately. Ammonia with formaldehyde forms hexamethylenetetramine and again decomposes with the consumption of formaldehyde; therefore it was assumed that the initial increase of pH in the former case was due to formation of formic acid by oxidation of free formaldehyde, as when the reaction was carried out in absence of oxygen the pH did not decrease. Cations such as NH_4^+ , Na^+ , K^+ , and Ba^{++} probably act as oxygen-carriers in the oxidation of formaldehyde to formic acid.

II—Reaction of urea with formaldehyde proceeds in two stages, viz. addition and condensation. The two components were treated in alkaline media of several different molecular ratios and strengths, and the rate of addition and condensation reactions was determined by measuring the hydroxymethyl group and free formaldehyde. The addition reaction proceeds continuously, while condensation after a time reaches a constant state. It is assumed that H^+ acts as a negative catalyst for the addition reaction, combining with the unshared electron pair of the N atom in urea and preventing the approach of the C atom in formaldehyde; therefore, removal of H^+ accelerated the addition reaction, but OH^- is regarded as an indirect catalyst for both the addition and condensation reactions. The difference between the hydroxymethyl and the methylene condensations is discussed.

III—The reaction velocity constant k and the activation energy A of the addition reaction were calculated for the urea-formaldehyde reaction under different conditions. Because urea is a bifunctional compound its concentration in a calculation of k must be multiplied by a factor n . In alkaline and neutral media $n = 2$, and at pH 4, where methylene condensation did not occur, $n = 1$; the uniformity of calculated values of k was best in neutral medium, where the pH remained constant throughout. $A = 11.6$ kcal. in alkaline medium and 9.5 kcal. in acid medium, indicating that the addition reaction proceeds much more quickly in the former. C. O. C.

Degradation and Weathering of Plastics. A. R. Burgess. *Chem. and Ind.*, 78-81 (26 Jan. 1952).

Aspects of oxidative, thermal, and photo-degradation of polymers, inhibition of degradation, relation of theory to weathering, and the development of accelerated tests are discussed. J. W. B.

N-Substitution in Polyamides. J. R. Lewis and R. J. W. Reynolds. *Chem. and Ind.*, 958-961 (10 Nov. 1951).

The effects of N-alkylation and N-alkoxymethylation of polyamides on their physical properties are discussed. J. W. B.

Development of Coumarone Resins as Raw Material for the Lacquer and Dye Industries.

H. Wille, *Brennstoff-Chem.*, 32, 238-243 (1951); *Chem. Abs.*, 45, 10,658 (25 Nov. 1951).

An account of the production of the coumarone-indene fraction from light oil and of its testing. Data are given of the amounts of these resins used in German industry; American production is much greater. C. O. C.

PATENTS

Polymerising or Condensing Chemical Substances.

A. A. Brach, *BP* 665,282

Many polymerisations or condensations may be carried out by irradiating the monomer or components with electrons having a velocity equivalent to > 500 kv. Often there is no need to use either heat, pressure, or catalysts, but in the case of melamine-formaldehyde monomers it is preferable to carry out the process at 30°C. in order to obtain a product of more uniform mol. wt. C. O. C.

Protecting Rubber against Sunlight.

Standard Oil Co., *USP* 2,561,671

Addition to rubber of the polymeric residue produced when liquid hydrocarbons and oxygenated compounds are produced from H_2 and CO in presence of an Fe catalyst confers resistance against deterioration on exposure to sunlight. C. O. C.

Silicon-containing Rubber Compositions.

Dow Corning Corp., *USP* 2,561,177

The hydro-polyalkoxysilyl addition products of natural rubber, gutta percha, balata, and elastomeric butadiene-styrene copolymers, which addition products contain $SiR'(O-R^2)_2$ groups (R^1 = methyl or phenyl; R^2 = methyl, ethyl, or propyl) bonded to carbon radicals of the rubber or other polymer by carbon-silicon linkages, are miscible with organic elastomers. They can be used as waterproofing agents, varnishes, and film-forming materials. They may be applied in solution to, and hydrolysed in contact with, cellulosic materials. Paints are obtained by mixing them with a pigment. C. O. C.

Linoleum Composition.

Armstrong Cork Co., *USP* 2,561,427

Addition of a hydrolysed vinyl chloride-vinyl acetate copolymer to linoleum compositions results in decided reduction in the maturing time. Linoleum made from such compositions has increased resiliency, water and alkali resistance, flexibility, and toughness. C. O. C.

Liquefying Highly Polymerised Vinyl Acetate.

A. Dawant, *BP* 665,758

Highly polymerised vinyl acetate can be used as a coating composition at 140°C. without use of a solvent if there has been incorporated with it a resin acid and a chlorinated hydrocarbon. C. O. C.

Coloured Nylon Articles.

Polymer Corp., *USP* 2,558,992

The nylon is rendered opaque, e.g. by incorporating TiO_2 , before it is extruded or moulded. Then, after it has been shaped, it is dyed. This results in uniform coloration of the article irrespective of the varying thickness of portions of it. C. O. C.

Polymeric Triazine Derivatives.

American Cyanamid Co., *BP* 665,191

The polymers of compounds of formula—



(A = a univalent sat. aliphatic, aromatic, or nuclearyl halogenated aromatic hydrocarbon radical or a radical of formula NR^1R^2 (R^1 and R^2 = H or the foregoing) or $O-R^2$ (R^2 = R^1 or R^3); R^1 = univalent residue of a primary ethylenically unsat. monohydric alcohol of 3-10 C; n = 1-3), e.g. polymeric triallyl cyanurate, are resins of wide uses, e.g. for treating textiles or paper, as components for coating compositions, etc. C. O. C.

Water-soluble Synthetic Resinous Compositions.

H. Honel, *BP* 665,195

Combination of (a) a water-soluble hardenable condensate, e.g. resol, (b) a liquid water-soluble plasticiser containing acidic and hydroxyl groups, and (c) ammonia or a water-soluble strong organic nitrogen base yields a water-soluble product suitable for lacquers, binding agents, impregnants, etc. C. O. C.

Ultraviolet Filtering Materials.

General Aniline, *BP* 665,371

Ultraviolet-absorbing compositions are formed by incorporating a uniform dispersion of 4-benzoylresorcinol into plastic transparent sheets. In one example benzoyl peroxide (50 mg.) and 4-benzoylresorcinol (200 mg.) are added to methyl methacrylate monomer (175 ml.), and the mixture is polymerised in a heated mould to a hard sheet $\frac{1}{8}$ in. thick. J. W. B.

Cation-modified Clays—Plastics, Pigments, etc.

National Lead Co., *BP* 665,474

A clay having base-exchange properties is treated first with an onium compound and then with an organic compound whose radical can replace an H atom of the organic portion of the onium radical. Various products may be made in this way, e.g. thermosetting and thermoplastic plastics, coloured pigments, reinforced rubbers, etc. C. O. C.

Crayon for Marking Rubber (V p. 132).

XIV—ANALYSIS; TESTING; APPARATUS

Estimation of Calcium in Water.

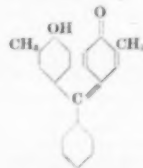
A. G. Knight, *Chem. and Ind.*, 1141 (22 Dec. 1951).

A modification of the method whereby small amounts of Ca and Mg are estimated by sequestration with disodium ethylenediaminetetra-acetate is described, using murexide (ammonium purpurate) indicator, which turns slowly from red to purple as Ca is sequestered; Naphthol Green B is used as a screen to assist the end point. Murexide (0.2 g.), Naphthol Green B (0.5), and pure NaCl (100) are ground together to give a uniformly coloured mixture, of which 0.2 g. is used to each 100 ml. of test solution. The colour changes from olive green \rightarrow darkens \rightarrow reddish grey just before end-point \rightarrow blue. This point is easily recognised with brief practice. J. W. B.

Determination of o-Tolyl Ester in Tritolyl [Tricresyl] Phosphate.

J. Haslam and D. C. M. Squirell, *Analyst*, 77, 71-74 (Feb. 1952).

Commercial tritolyl (tricresyl) phosphate is a mixture of isomers, of which the m- and p-forms are harmless but the o-isomer is highly toxic. Cases have been reported in Germany of paralysis caused by contact with vinyl sheet plasticised with tritolyl phosphate too rich in o-isomer. Legal limits to the content of this constituent have been set in Germany (6%) and Sweden (3%), and one of 3% is being considered in the U.S.A. Determination of the o-tolyl ester is based upon its reaction with benzaldehyde (freshly prepared) to give a dye which is red in acid soln., and blue-violet in alkaline soln. It has the following constitution—



The m- and p-isomers yield only colourless products. The mixture of isomers is hydrolysed with Cellulosolve potash, extracted with ether (on the alkaline side) to remove interfering substances, and then acidified and extracted with ether to remove the cresols. These are distilled off and condensed with benzaldehyde under carefully defined conditions. After purification, the condensation product is estimated in methanol soln. using the Spekker. Full details of procedure are given. Phenol and 2:6-xyleneol interfere, but not seriously. J. W. D.

Determination of Wetting Power. G. Reutenauer, J. P. Bailey, and S. Dupin. *Amer. Dyestuff Rep.*, 41, P 25-P 30 (7 Jan. 1952).

The hydrometer method of determining wetting power is described and discussed. It is used with either cotton skeins or strips of cotton fabric, which are either attached to or hung from a hydrometer; density readings vary according to penetration of the material by the wetting solution, and results accord well with those obtained by other methods. Precision is superior, and results may be expressed in a more scientific manner, and are in direct proportion to the elimination of air from the fabric.

J. W. B.

Progress in Wetting and Rewetting Test Methods. L. Shapiro. *Amer. Dyestuff Rep.*, 41, P 16-P 24 (7 Jan. 1952).

Rate of wetting measurements are made on cotton by the hydrometer method, and an attempt is made to correlate the curves obtained with those from weighted tape and floating square tests. Factors leading to anomalous results, such as air entrapment and escape of air bubbles from cut and uncut tapes, are discussed. The effects of drying and baking on rewetting are investigated, and it is believed that partial cleansing of the cotton surface may contribute to superior rewetting. Reproducible results are obtained by the hydrometer method.

J. W. B.

Studies on Dye Analyses. I—Detection of Annatto in Food Fats. H. Thaler and R. Scheler. *Z. Lebensmitteluntersuch. u. Forsch.*, 93, 220-224 (1951); *Chem. Abs.*, 46, 658 (25 Jan. 1952).

A chromatographic method is used, bixin even in presence of fat being strongly adsorbed by standard Al_2O_3 . Other carotenoids that may be present in food fats as well as most synthetic dyes are not retained by the oxide. Martius Yellow and Sudan G (aniline-azoresorcinol) are adsorbed similarly to bixin, but unlike it are eluted with alcohol. The Carr-Price reaction (*Chem. Abs.*, 20, 3020) can be used for confirmation. Excessive amounts of free fatty acid inhibit adsorption of bixin.

C. O. C.

Volumetric Estimation of Nitro Groups in Cellulose Nitrate. R. Leclercq and J. Mathé. *Bull. Soc. chim. Belg.*, 60, 296-300 (July-Aug. 1951).

A soln. of cellulose nitrate in conc. H_2SO_4 at 0°C. is titrated with ferrous ammonium sulphate to a mauve-colored end point. It is claimed that the method combines simplicity with rapidity and precision. Data are also provided to show that cellulose nitrate undergoes a transition in H_2SO_4 soln. which is a function of temperature and time.

G. L.

A Simple Solution to the Artificial Light Problem in Colour Matching. H. R. Davidson. *Amer. Dyestuff Rep.*, 41, 1-10 (7 Jan. 1952).

Dyes for colour matching in two- or multi-colour mixtures are selected so that the product will change in shade under varying types of illumination in exactly the same manner as the pattern. With the aid of a spectrophotometer and a file of spectrophotometric curves the correct composition can be calculated in about 2 hr. by given mathematical formulae; trial dyeings are then followed by comparatively short corrections. An example of a five-colour match is presented in detail, and it is claimed that much time can be saved compared with conventional methods.

J. W. B.

Rapid Control Test to Indicate Colour Fastness to 105°F. Washing. A.A.T.C.C. *Amer. Dyestuff Rep.*, 41, P 34 (21 Jan. 1952).

The test solution is prepared by adding slowly carboxymethyl cellulose (12.5 g.) to nearly boiling water (250 ml.), with stirring, standing for 15 min., and adding neutral soap (2.5 g.) in warm water (250 ml.). The test fabric (3 in. x 3 in.) is placed on a glass plate (3 in. x 6 in.), and test solution (0.5 ml.) pipetted on it; the spotted specimen is covered with a blotter, and a hot flat-iron (121°C.) applied for 5 min. Appearance of colour on the blotter indicates that colour would bleed into solution and probably stain adjacent uncoloured fabric in the No. 1 Wash Test.

J. W. B.

Testing of Water-repellent Finishes on Textiles. H. M. Ulrich. *Textil Rund.*, 6, 548-552 (Dec. 1951).

A scheme for the evaluation of impregnated fabrics is provided. Methods for the removal of hydrophobic substances from wool and cellulose fibres are described.

Tests for the identification of fats, soaps, silicones, and esterified or etherified celluloses are given. A spot test for Zr salts is based on the formation of a red-violet slizarin lake insol. in dil. HCl.

G. L.

PATENTS

Wear Tester. Orr Felt & Blanket Co. *USP* 2,561,133

A machine especially suitable for use on papermakers' felts and similar fabrics has two knurled frictional rolls working in conjunction with a material-carrying roll. All three rolls rotate at the same number of r.p.m., the frictional effect being obtained by making the rolls of different diameters. The rolls are so arranged that engagement of the fabric between one of the knurled rolls and the material-carrying roll causes tension in one direction, while engagement with the other knurled roll creates tension in the opposite direction.

C. O. C.

Phase Microscopy. F. Zernike. *BP* 664,593

A better image is obtained by changing the phase of part of the light rays which co-operate to form the image. This can be done by using a plate made up of a transparent strip embedded in a transparent material, both components being chosen to secure a desired law of variation of the difference in their refractive indices.

C. O. C.

Producing Phase Contrasts when Examining Microscopic Objects. E. Leitz. *BP* 665,944

The device comprises an objective and a condenser for forming an annular base of illumination of such small width that the broadening (due to diffraction of the light rays at the entrance pupil of the objective) of the annular image of the base formed in the exit pupil of the objective is perceptible. The objective has means other than an ordinary phase plate for creating a phase difference between light not diffracted by the object and light diffracted by the object when the distance between the light base and the objective has been suitably adjusted.

C. O. C.

Indicators for Judging Colours. I. & R. Morley. *BP* 664,863

A device for indicating the hue which will be exhibited when very fine-mesh fabrics, e.g. stockings, are worn comprises a transparent solid sheet having alternate transparent and coloured zones, so selected and arranged that when the device is viewed against a flesh-coloured background it reveals the hue corresponding to that which will be shown by the material when worn.

C. O. C.

XV— MISCELLANEOUS

Photographic Image on Anodised Aluminium. N. I. Kirillov and A. S. Khaynman. *J. Appl. Chem. U.S.S.R.*, 24, 1019-1025 (Oct. 1951).

The processes of anodising aluminium and of depositing sensitive silver compounds in the pores of the resulting oxide film are examined, and conditions for producing satisfactory sensitive material are established. Also, diazotype sensitisation can be used. For this method, the oxide film, produced by anodic oxidation in sulphuric or chromic acid, should have a thickness of 10-15μ. The anodised plate is immersed for 5-10 min. in a sensitising soln. containing R salt, diazotised p-dimethylaniline, and phosphoric acid, and it is then drained and dried. Development is by exposure to ammonia fumes. The resultant blue or violet images on a lustrous background are very effective.

A. E. 8.

PATENTS

Colouring Margarine. E. F. Drew & Co. *USP* 2,559,411

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C. O. C.

Recovering Streptomycin from its Orange II or Naphthol Blue Black Salt. Chas. Pfizer & Co. *USP* 2,560,889-90

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C. O. C.



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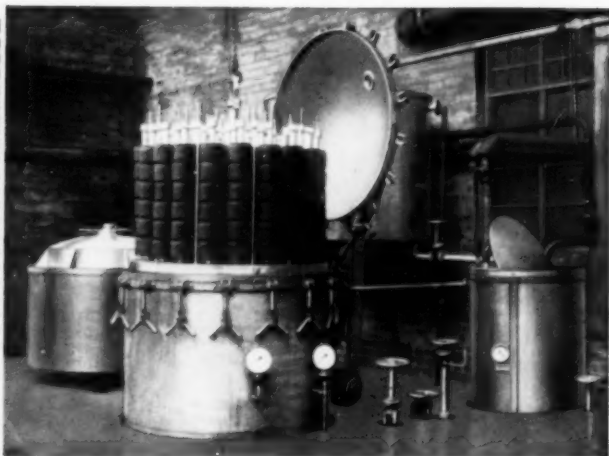
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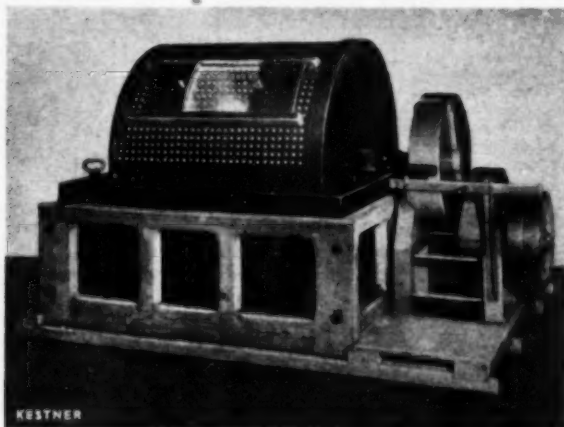
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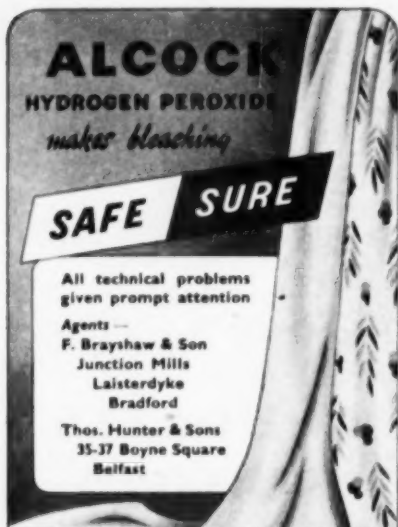
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